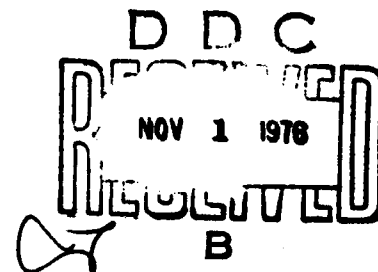AD A031436

MOISTURE ABSORPTION AND DESORPTION OF COMPOSITE MATERIALS

THE UNIVERSITY OF MICHIGAN
MECHANICAL ENGINEERING DEPARTMENT
ANN ARBOR, MICHIGAN 48109

JUNE 1976

ANNUAL REPORT MARCH 1975 - MARCH 1976

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Project Monitor

FOR THE DIRECTOR

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Nonmetallic Materials Division

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFML-TR-76-102	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) MOISTURE ABSORPTION AND DESORPTION OF COMPOSITE MATERIALS		5. TYPE OF REPORT & PERIOD COVERED Technical Annual Mar 1975-Mar 1976
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) George S. Springer Chi-Hung Shen		8. CONTRACT OR GRANT NUMBER(s) F33615-75-C-5165 NEW
9. PERFORMING ORGANIZATION NAME AND ADDRESS The University of Michigan Mechanical Engineering Department Ann Arbor, Michigan 48109		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 734003A5
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Materials Laboratory (AFML/MBM) Air Force Wright Aeronautical Laboratories Wright-Patterson Air Force Base, Ohio 45433		12. REPORT DATE June 1976
		13. NUMBER OF PAGES 59
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Moisture Absorption and Desorption
Diffusivity
Maximum Moisture Content
Graphite Epoxy Composites

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Expressions are presented for the moisture distribution and the moisture content as a function of time of one dimensional homogeneous and composite materials exposed either on one side or on both sides to humid air or to water. The results apply during both moisture absorption and desorption when the moisture content and the temperature of the environment are constant. Test procedures are described for determining experimentally the values of the moisture content and the diffusivity of composite materials. A series of tests using unidirectional and [   ] Graphite T-300 Fiberite 1034 composites were

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performed in the temperature range 300-425 K with the material submerged both in moist air (humidity 0 to 100%) and in water. The test data support the analytical results and provide the moisture absorption and desorption charac- teristics of such composites. Extension of the results to materials exposed to time varying environmental conditions is indicated.

Attempts were made to correlate the moisture content of the material with a) changes in electric resistance and b) changes in the hardness of the material. The latter method promises to provide an indication of the moisture content.

FOREWORD

This annual report was submitted by Dr. George S. Springer and Dr. Chi-Hung Shen of The University of Michigan, Mechanical Engineering Department, Ann Arbor, Michigan, under contract F33615-75-C-5165, Project 7340, Task 734003, with the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Stephen W. Tsai, AFML-MBM was the laboratory project monitor.

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x

LIST OF SYMBOLS

a   constant (dimensionless)

A   area (mm^2)

b   constant (dimensionless)

B_K parameter defined by eq. (15) (dimensionless)

B_D parameter defined by eq. (16) (dimensionless)

c   moisture concentration in the material $(g\ mm^{-3})$

c_a ambient moisture concentration $(g\ mm^{-3})$

C   specific heat of the material $(Jg^{-1}\ K^{-1})$

D   mass diffusivity $(mm^2\ s^{-1})$

G   parameter defined by eq. (3) (dimensionless)

h   thickness of specimen (mm)

K   thermal conductivity $(W\ mm^{-1}\ K^{-1})$

l   length of specimen (mm)

m   moisture content in the material (N)

M   percent moisture content in the material (dimensionless)

n   width of specimen (mm)

s   equivalent thickness of specimen, see Fig. 2 (mm)

t   time (s)

t_L time defined in Fig. (5) (s)

t^* dimensionless time defined in Fig. (2) (dimensionless)

T   temperature (K)

v_f volume fraction of fiber (dimensionless)

W   weight of material (N)

W_d dry weight of material (N)

Greek letters

        fiber orientation with respect to the x, y, and z axes,

        see Fig. 1 (rad)

        percent relative humidity (dimensionless)

        density of material (g mm^{-3})

Subscripts

f	fiber
i	initial state
m	maximum saturation
r	matrix
x,y,z	in the x, y, and z directions
11	in the direction parallel to the fiber
22	in the direction normal to the fiber

SECTION I

INTRODUCTION

Owing to their favorable performance characteristics composite materials have been gaining wide use in commercial, military, and space applications. There is some concern, however, that the mechanical properties of such materials may suffer when the material is exposed to moisture for long periods of time. Therefore, in order to utilize the full potential of composite materials their response to moist environments must be known. The objective of this investigation was to determine the moisture content as a function of time of composite materials when the material is either exposed to humid air or fully submerged in water. Although the main interest in this investigation was in the moisture absorption and desorption of composite materials, the results also apply to homogeneous materials.

First, results are presented which apply directly for single layered composites when the moisture content and the temperature of the environment are constant (Sections II-VII). The major conclusions obtained for this problem were described by the authors in The Journal of Composite Materials (Reference 1). The results thus obtained are also needed for calculating moisture contents of single or multilayered composites when the ambient conditions vary with time (Section VIII).

SECTION II

THE PROBLEM

Consider a plate of thickness h exposed on two sides to the same moist environment (Fig. 1a). The plate is taken to be infinite in the y and z directions so that the moisture content and the temperature inside the plate vary only in the x direction (i.e. the problem is one dimensional). Initially (time $t < 0$) the temperature T_i and the moisture concentration c_i inside the plate are uniform. The plate is suddenly exposed to a moist environment in which the temperature T_a and moisture concentration c_a are constant. The objective is to determine the temperature distribution T, the moisture distribution c, and the total moisture content m of the material as a function of time.

The temperature and the moisture concentration are described by the Fourier and the Fick equations

$$\rho C \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} K_x \frac{\partial T}{\partial x} \tag{1}$$

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x} D_x \frac{\partial c}{\partial x} \tag{2}$$

and the initial and boundary conditions

$$\begin{aligned} T &= T_i \\ c &= c_i \end{aligned} \qquad 0 < x < h \qquad t \leq 0 \tag{3a}$$

$$\begin{aligned} T &= T_a \\ c &= c_a \end{aligned} \qquad x = 0 \,; x = h \qquad t > 0 \tag{3b}$$
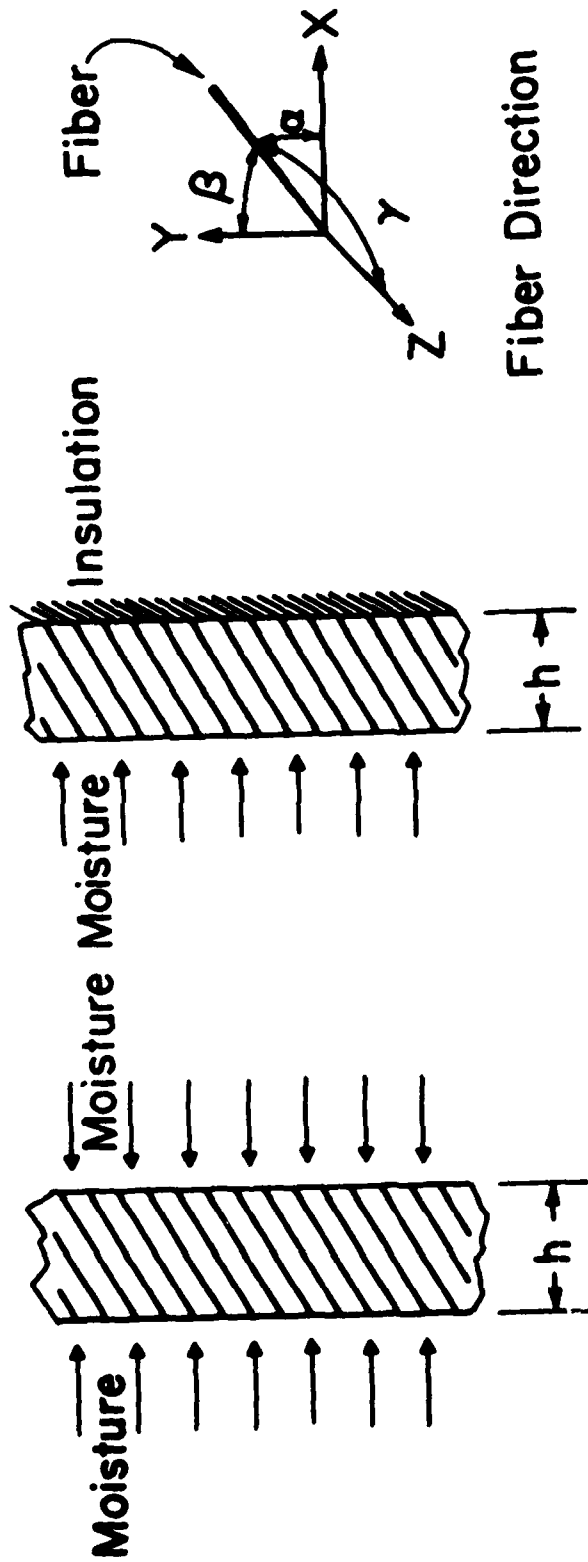
Figure 1. Description of the Problem

The moisture concentration at the surface of the material c_m is related to the moisture content of the environment.

The thermal diffusivity $K_x/\rho C$ and the mass diffusivity D_x depend on the temperature and on the moisture concentration and hence on the distance x. The parameters $K_x/\rho C$ and D_x are a measure of the "speed" by which the temperature and the moisture concentration change inside the material. For most materials the ratio $(K_x/\rho C)/D_x$ is of the order of 10^6. Thus, the temperature approaches equilibrium about 10^6 times faster than the moisture concentration. For example, for a 12.5 mm thick Graphite T-300 Fiberite 1034 composite exposed to 90 percent humid air at 350 K the temperature becomes nearly uniform across the plate in about 15 seconds while the moisture concentration reaches equilibrium only in about 13 years. Therefore, in most practical situations the temperature inside the material may be taken to be *the same as the ambient temperature.* It has also been observed that the diffusivity changes very little with the moisture content (Reference 2). With the temperature and the diffusivity being constant inside the material the problem is described by the equations

$$\frac{\partial c}{\partial t} = D_x \frac{\partial^2 c}{\partial x^2} \tag{4}$$

$$c = c_i \qquad 0 < x < \ell \qquad t \leq 0 \tag{5a}$$

$$c = c_a \qquad x = 0 \; ; \; x = \ell \qquad t > 0 \tag{5b}$$

SECTION III

MOISTURE CONTENT

The moisture distribution and the weight of moisture inside the material is obtained from equations (4) and (5). The solution of these equations is (Reference 3)

$$\frac{c - c_i}{c_m - c_i} = 1 - \frac{4}{\pi} \sum_{j=0}^{\infty} \frac{1}{(2j+1)} \sin \frac{(2j+1)\pi X}{h} \exp\left[-\frac{(2j+1)^2 \pi^2 D_x t}{h^2}\right] \tag{6}$$

The total weight of the moisture in the material is obtained by integrating equation (6) over the plate thickness

$$m = \int_0^h c g \, dx \tag{7}$$

The result of this integration is (Reference 3)

$$G \equiv \frac{m - m_i}{m_m - m_i} = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp\left[-(2j+1)^2 \pi^2 \left(\frac{D_x t}{h^2}\right)\right]}{(2j+1)^2} \tag{8}$$

m_i is the initial weight of the moisture in the material (i.e. the weight prior to exposure to the moist environment) and m_m is the weight of moisture in the material when the material is fully saturated, in equilibrium with its environment.

The time (t_m) required for a material to attain at least 99.9 percent of its maximum possible moisture content can be determined from equation (8) and is given as

$$t_m = \frac{0.67 \, h^2}{D_x} \tag{9}$$

Equation (8) may be approximated by the expression (see Figure 2)

$$G = 1 - \exp\left[-0.73\left(\frac{D_x t}{h^2}\right)^{0.75}\right] \tag{10}$$
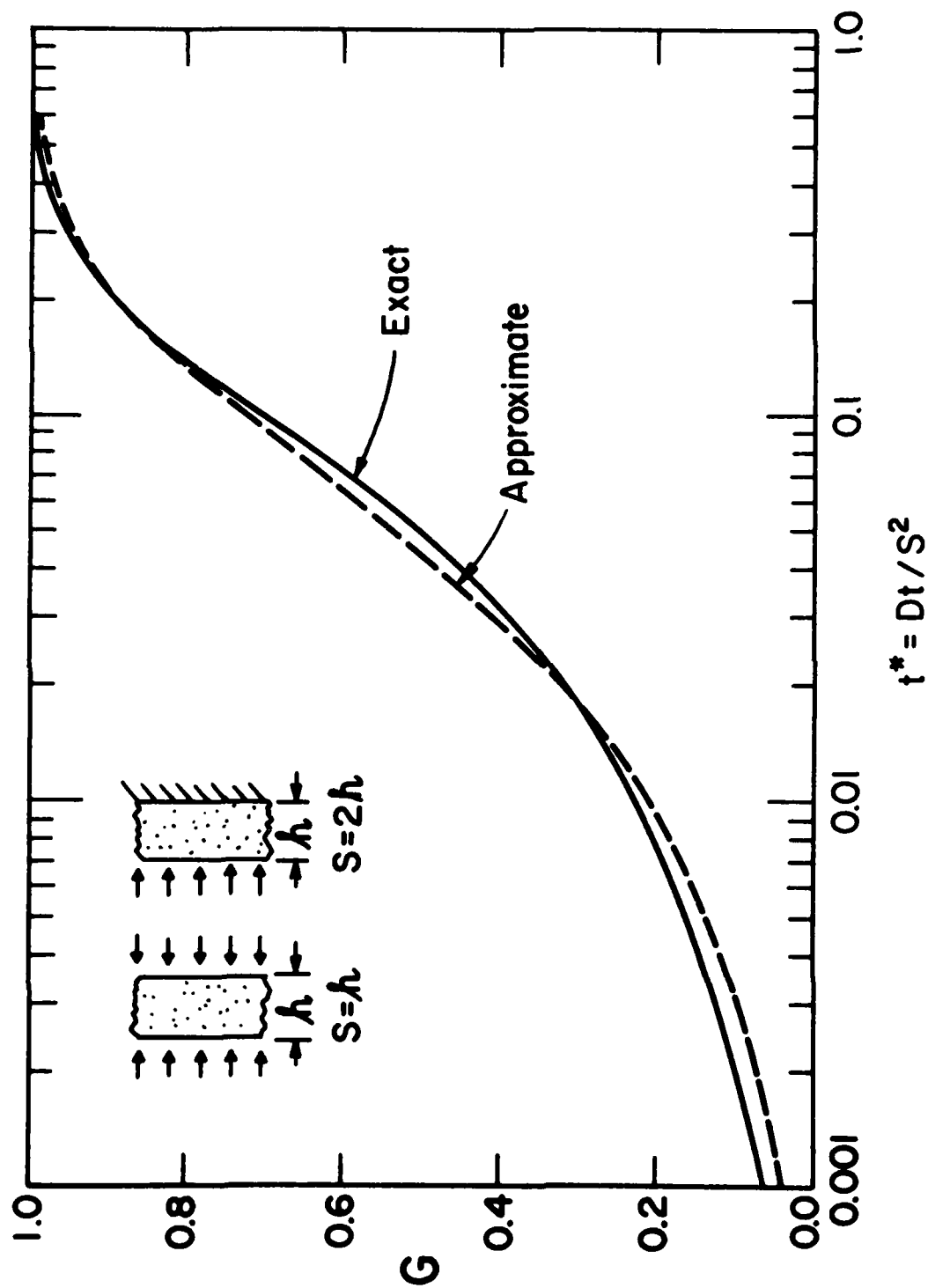
Figure 2. The Variation of the Parameter G with the Dimensionless Time $t^* = Dt/s^2$. Exact: Eq. (8); Approximate: Eq. (10)

In most practical situations we are interested in the percent moisture content as manifested by the weight gain of the material. Thus, in practice the parameter of interest is the percent moisture content defined as

$$M = \frac{\text{WEIGHT OF MOIST MATERIAL} - \text{WEIGHT OF DRY MATERIAL}}{\text{WEIGHT OF DRY MATERIAL}} \times 100 = \frac{W - W_d}{W_d} \times 100 \quad (11)$$

By noting that

$$W = W_d + m \quad (12)$$

equation (8) may be rearranged in the form

$$M = G(M_m - M_i) + M_i \quad (13)$$

Equation (13) applies to both absorption and desorption. Furthermore, it can be used when the material is exposed to the environment either on one or on two sides. The use of the foregoing results is illustrated by the following examples.

Example 1. A 12.5 mm thick Graphite T-300 Fiberite 1034 plate is exposed on both sides to air at temperature 350 K and 90 percent humidity. The plate is of $\pi/4$ construction with all fibers being parallel to the surface of the plate ($\alpha = 90°$). The initial moisture content of the plate is 0.5 percent. Estimate the time required to reach one percent moisture content. From equations (10) and (19)

$$t = \frac{s^2}{D_x} \left[-\frac{1}{7.3} \ln \left(1 - \frac{M - M_i}{a \phi^b - M_i} \right) \right]^{1/0.75} \quad (14)$$

For graphite T-300 Fiberite 1034 ($v_f = 0.68$) the constants a and b are 0.00014 and 2, respectively. The diffusivity is (Figure 3 and equation 18)

$$D_x = D_{22} = 2.6 \times 10^{-7} \quad mm^2 \, s^{-1}$$

Thus, equation (14) gives

$$t = \frac{(12.5)^2}{2.6 \times 10^{-7}} \left[-\frac{1}{7.3} ln \left(1 - \frac{1 - 0.5}{0.00014(90)^2 - 0.5} \right) \right]^{1/0.75} = 7.6 \times 10^7 \, s = 2.4 \text{ years}$$

For 90 percent humidity the maximum possible moisture content of the plate is

$$M_m = 0.00014 \, (90)^2 = 1.13 \, \%$$

The time required to reach at least 99.9 percent of this moisture content is (equation 9)

$$t_m = \frac{0.67 \times (12.5)^2}{2.6 \times 10^{-7}} = 4 \times 10^8 \, s = 12.8 \text{ years}$$

If the plate were exposed to the environment on one side only the time required to reach one percent moisture content would be

$$t = \frac{(2 \times 12.5)^2}{2.6 \times 10^{-7}} \left[-\frac{1}{7.3} ln \left(1 - \frac{1 - 0.5}{0.00014(90)^2 - 0.5} \right) \right]^{1/0.75} = 3.0 \times 10^8 \, s = 9.6 \text{ years}$$

and the time required to reach at least 99.9 percent of the maximum possible moisture content would be

$$t_m = \frac{0.67 \times (2 \times 12.5)^2}{2.6 \times 10^{-7}} = 1.6 \times 10^9 \, s = 51 \text{ years}$$

Example 2.   The initial moisture content of the plate specified in the above example is 1 percent. The plate is then exposed on both sides to humid air of temperature 333 K and relative humidity 10 percent. Estimate the moisture content of the plate after 10 days.

From equations (10) and (13)

$$M = \left\{ 1 - exp \left[-7.3 \left(\frac{10 \times 24 \times 3600 \times 1.13 \times 10^{-7}}{12.5 \times 12.5} \right)^{0.75} \right] \right\} [0.00014(90)^2 - 1] + 1 = 0.97 \, \%$$

Thus, in 10 days the moisture content would be reduced from one percent to 0.97 percent.

SECTION IV

DIFFUSIVITY

The diffusivity D_x can be measured directly. For composite materials the measurements must be performed properly taking into consideration the anisotropy of the material. For fiber reinforced composites the diffusion coefficient D_x can also be calculated from the following parameters: a) the diffusivity of the matrix D_r, b) the diffusivity of the fibers D_f, c) the volume fraction of the fiber v_f, and d) the orientation of the fibers with respect to the exposed surface (α, β, γ , Figure 1). The relationship between D_x and D_m, D_f, v_f, and the fiber orientation can be established by noting the similarities between heat conduction and moisture diffusion through the material (equations 1 and 2). Approximations for the thermal conductivities parallel and normal to the fibers were given by Springer and Tsai (Reference 4) ($v_f < 0.785$)

$$K_{//} = (1 - v_f) K_r + v_f K_f \tag{15a}$$

$$K_{22} = (1 - 2\sqrt{v_f/\pi}) K_r + \frac{K_r}{B_k} \left[\pi - \frac{4}{\sqrt{1 - (B_k^2 v_f/\pi)}} \tan^{-1} \frac{\sqrt{1 - (B_k^2 v_f/\pi)}}{1 + \sqrt{B_k^2 v_f/\pi}} \right]$$

$$B_k \equiv 2 \left(\frac{K_r}{K_f} - 1 \right) \tag{15b}$$

Analogously, we can write

$$D_{//} = (1 - v_f) D_r + v_f D_f \tag{16a}$$

$$D_{22} = (1 - 2\sqrt{v_f/\pi}) D_r + \frac{D_r}{B_D} \left[\pi - \frac{4}{\sqrt{1 - (B_D^2 v_f/\pi)}} \tan^{-1} \frac{\sqrt{1 - (B_D^2 v_f/\pi)}}{1 + \sqrt{B_D^2 v_f/\pi}} \right]$$

$$B_D \equiv 2 \left(\frac{D_r}{D_f} - 1 \right) \tag{16b}$$

Generally, the diffusivity of the fiber is small compared to the diffusivity of the matrix ($D_f \ll D_r$), and equations (16a) and (16b) reduce to ($v_f < 0.785$)

$$D_{//} = (1 - v_f^2) D_r \tag{17a}$$

$$D_{22} = (1 - 2\sqrt{v_f/\pi}) D_r \tag{17b}$$

D_x is related to D_{11}, D_{22}, and D_r by the expressions (Reference 5)

$$D_x = D_{//} \cos^2 \alpha + D_{22} \sin^2 \alpha \tag{18}$$

$$D_x = D_r [(1 - v_f) \cos^2 \alpha + (1 - 2\sqrt{v_f/\pi}) \sin^2 \alpha] \tag{19}$$

In order to assess the validity of equation (19) the diffusivities of Graphite T-300 Fiberite 1034 composites were measured at different temperatures, and the measured values were compared to values calculated from equation (19). The tests were performed with 101.6 mm long, 12.7 mm wide, and 1.27 mm thick specimens (volume fraction v_f = 0.68, angle α = 90 degrees) according to the procedures outlined in the next section. Diffusivities of the pure matrix D_r were also measured with 50.8 mm x 12.7 mm x 1.27 mm specimens. The results for D_r are presented in Figure 3. In this figure the data of Hertz (Reference 6) is also included.

Comparison between the measured and calculated D_x values are shown in Figure 3. As can be seen from this figure there is good correlation between the measured and calculated diffusivities lending confidence to the validity of equation (19).
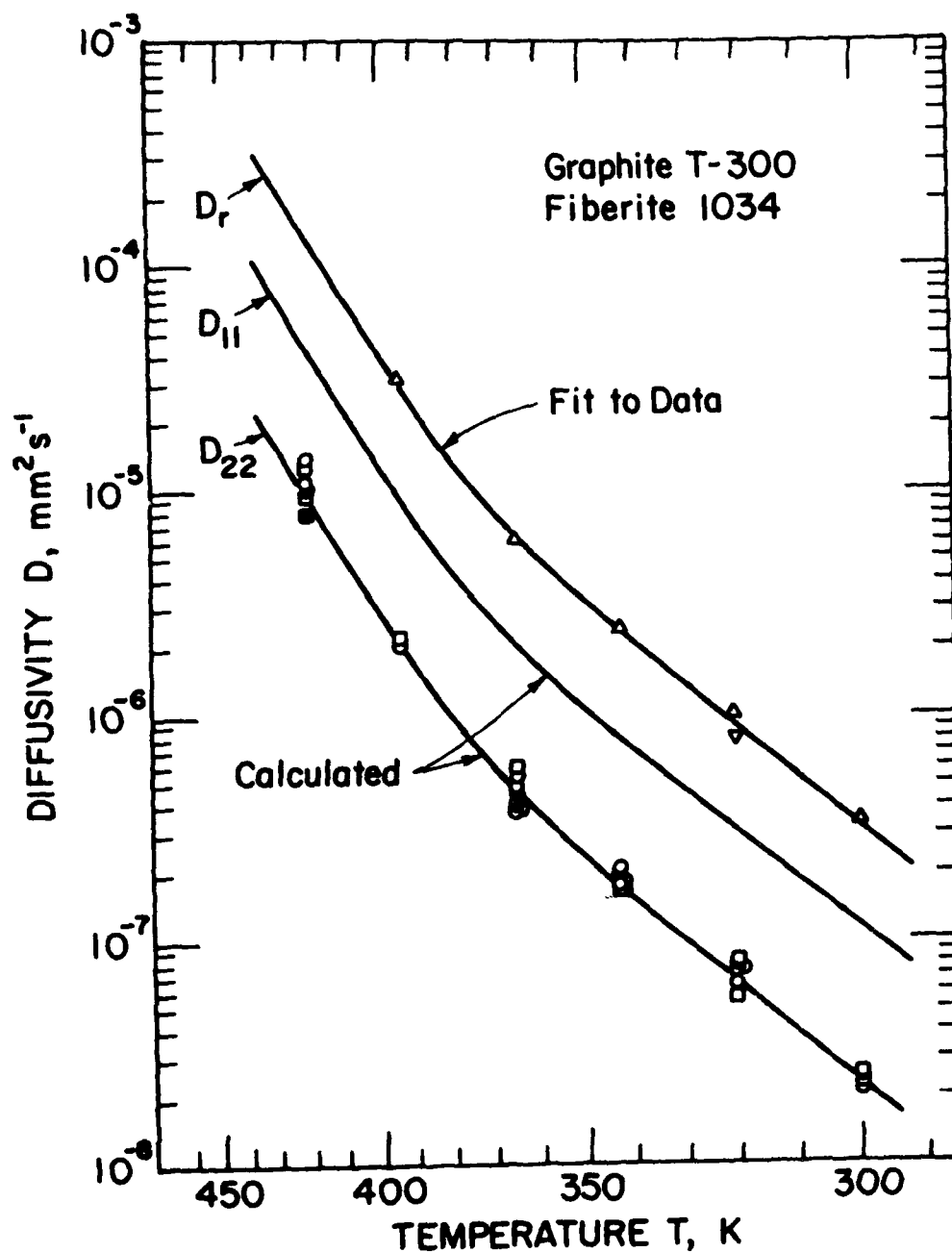
Fig. 3. The Matrix Diffusivity D_r, the Transverse Diffusivity D_{22}, and the Longitudinal Diffusivity D_{11} of Graphite T-300 Fiberite 1034 Composites. (D_{11} and D_{22} are for $v_f = 0.68$ and $\alpha = 90°$) o,●:Unidirectional; □ ,■:$\pi/4$ (Empty and Dark Symbols Represent Absorption and Desorption, Respectively. Δ : Present Data for Pure Matrix; ∇ : Data from Hertz [6]. Calculated D_{11} and D_{22} Curves are from Eqs. (17a and 17b).

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SECTION V

TEST PROCEDURES

In order to predict the moisture content of a material the maximum mois-
ture content M_m and the diffusivity D_x must be known (see equations 8 and 13).
The following test procedures may be used to determine these parameters:

1) The test specimen is made in the form of a thin plate $(h/l \ll 1$ and $h/n \ll 1$,
Figure 4), so that moisture enters predominantly through the nl surfaces of
the plate. A coating impermeable to moisture should be applied to the edges
of the plates (surfaces nh and lh). Metal coated tapes were found to provide
the best protection.

In the absence of coating a correction must be made for the moisture
entering through the edges (see Appendix A). For unidirectional composites
the fibers may be oriented in any arbitrary direction; specimens with fibers
normal ($\alpha = 0°$) or parallel ($\alpha = 90°$) to the nl surface are most convenient.
For laminated composites the fibers in each laminate must be parallel ($\alpha = 90°$) to the nl surface.

2) The specimen is completely dried in a desiccator and its dry weight W_d
is measured.

3) The specimen is placed in a constant temperature, constant moisture en-
vironment and its weight W is recorded as a function of time.

4) The moisture content (percent weight gain) $M = (W-W_d)/W_d$ is plotted
versus $\sqrt{t}$, as illustrated in Figure 5.

5) The tests are repeated for different temperatures and different environ-
mental moisture contents.

The above procedure yields a series of curves similar to the one shown
in Figure 5. Initially (when $t < t_L$, Figure 5) all curves are straight lines,

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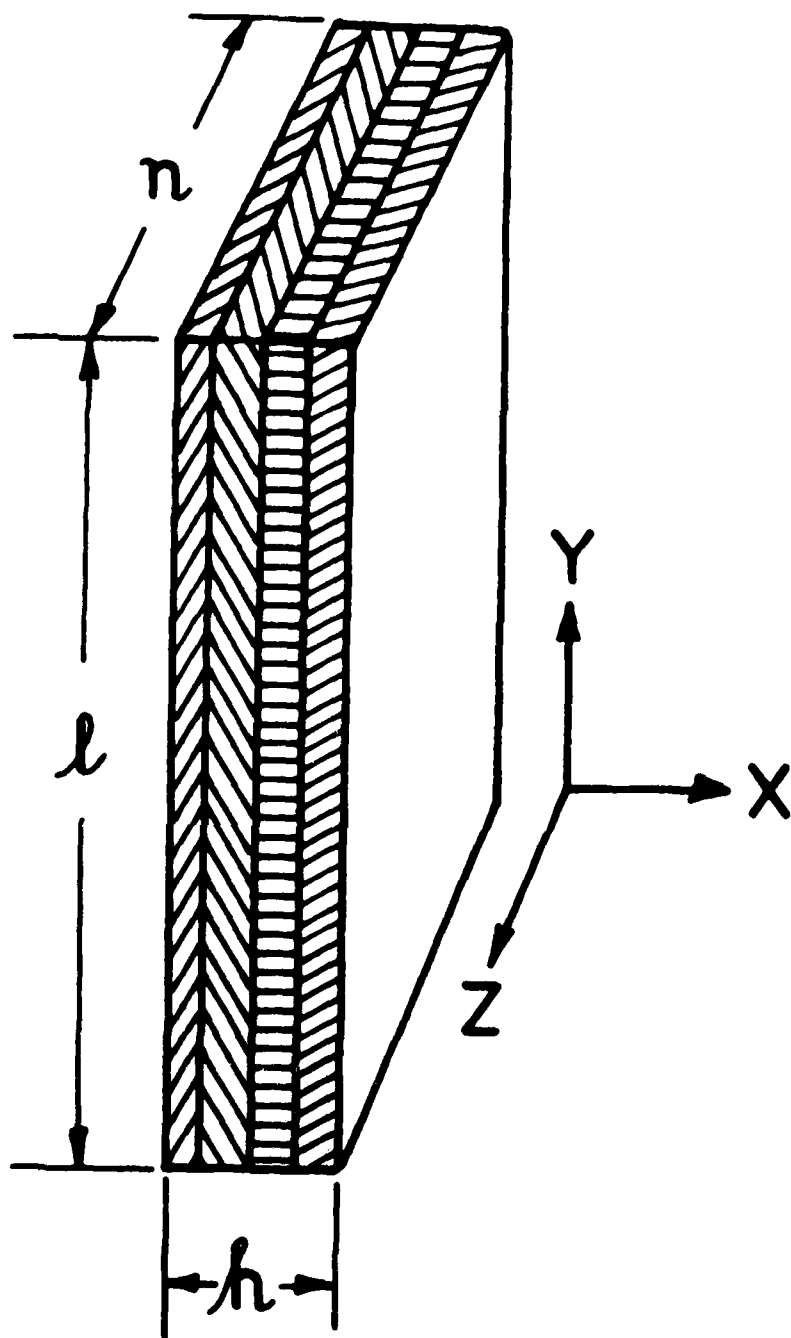
Figure 4. Geometry of the Test Specimen

MOISTURE CONTENT, M

M_m

M_2

M_1

$\sqrt{t_1}$ $\sqrt{t_2}$ $\sqrt{t_L}$ $\sqrt{t_m}$

SQUARE ROOT OF TIME, $\sqrt{t}$

Slope = $\dfrac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} = \dfrac{4 M_m}{h \sqrt{\pi}} \sqrt{D}$
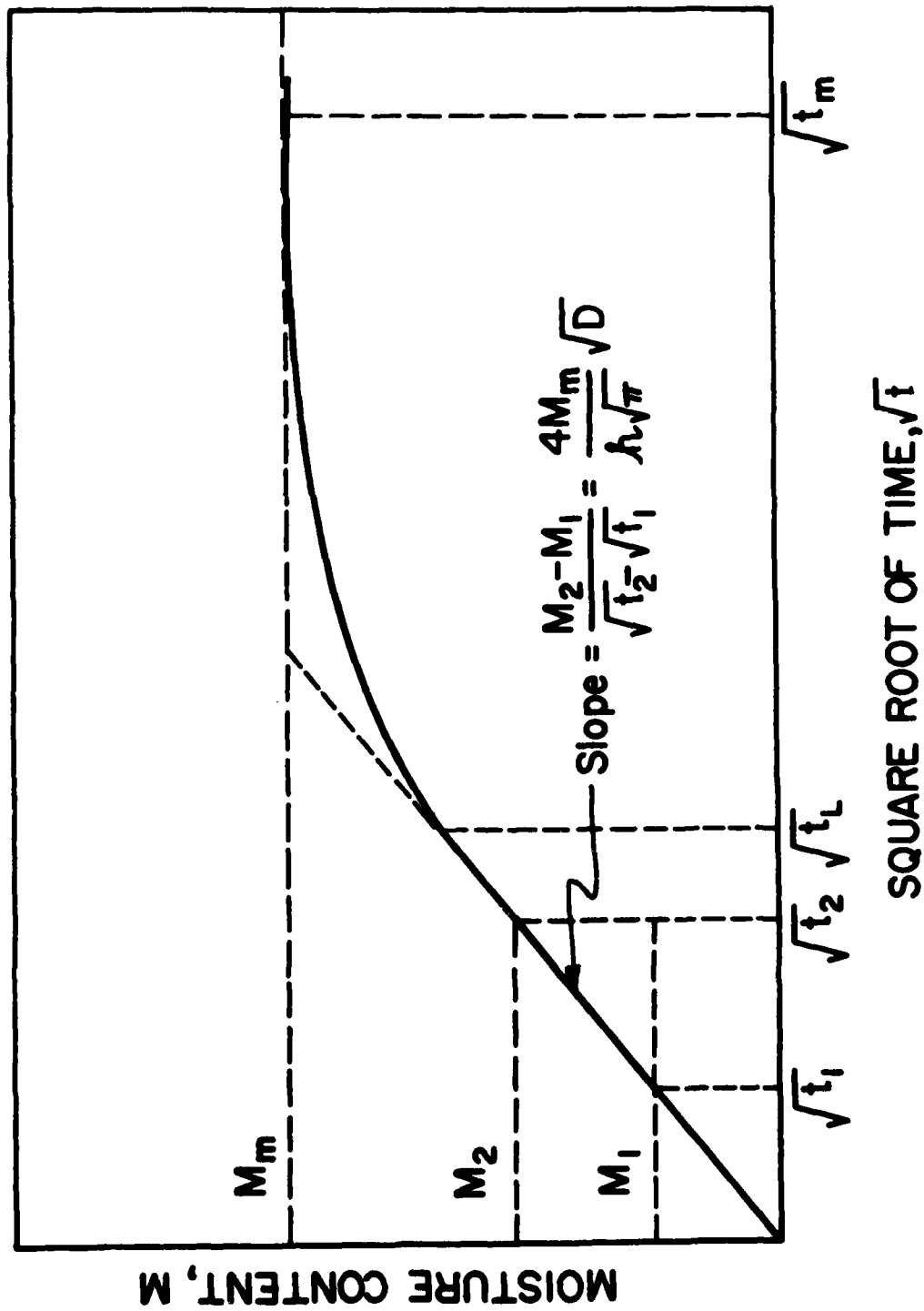
Fig. 5. Illustration of the Change of Moisture Content with the Square Root of Time. For $t < t_L$ the Slope is Constant.

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the slope being proportional to the diffusivity of the material. After a long period of time the curves approach asymptotically the maximum moisture content M_m. The value of M_m is a constant when the material is fully submerged in a liquid; it varies with the relative humidity when the material is exposed to moist air (Reference 7). In the latter case M_m may be expressed as

$$M_m = a \, \phi^b \tag{20}$$

The constants a and b are selected to provide the best fit to the data, as illustrated in the next section.

The diffusivity D is obtained from the initial slope ($t < t_L$) of the M_m versus $\sqrt{t}$ curve (see Appendix A)

$$D = \pi \left(\frac{h}{4 M_m} \right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2 \tag{21}$$

If the moisture entering the specimen through the "edges" (surfaces hn and hl, Figure 4) can be neglected D_x is

$$D_x = D \tag{22}$$

Including "edge effects", for a homogeneous material (Figure 4) D_x is (see Appendix A)

$$D_x = D \left(1 + \frac{h}{l} + \frac{h}{n} \right)^{-2} \tag{23}$$

For fiber reinforced composites D_x cannot be computed directly when the edge effects are taken into account. In this case D_x must be evaluated from the expressions derived in Appendix A.

If D_r is known then D_x can be obtained directly from equation (19). Hence only M_m must be obtained from the tests, in which case, the test times can be reduced by performing the tests at high temperatures.

The foregoing procedure requires that each specimen be tested until the maximum moisture content is reached ($t = t_m$). The tests may be accelerated by the following procedure.

1) Prepare a specimen in the form of a thin plate as described in point 1 above. The edges must be coated with a substance impermeable to moisture because the edge corrections cannot be applied now.

2) Measure the weight gain of a dry specimen ($M_i = 0$) as a function of time and plot M versus $\sqrt{t}$ beyond the point at which the slope of the curve is not a constant ($t > t_L$).

3) Assume a value for D_x.

4) Select a time t_1 so that $t = t_1 < t_L$.

5) Calculate G from either equation (8) or from Figure 2.

6) Calculate M_m for t_1 from equation (13) using the value of M measured at t_1.

7) Select a time t_2 so that $t = t_2 > t_L$.

8) Calculate G from either equation (8) or from Figure 2.

9) Calculate M_m for t_2 from equation (13) using the value of M measured at t_2.

10) Check whether M_m given by step 6 is equal to M_m given by step 9. If the two M_m values differ select a new diffusivity and repeat the procedure.

Using this procedure the tests can be terminated at a time t_2 which is much less than t_m.

For a layered composite, the average maximum moisture content $(M_m)_{avg}$ and the average diffusivity $(D_x)_{avg}$ can be estimated from the maximum moisture content and diffusivity of each layer. If the maximum moisture contents and diffusivities of layers of thicknesses L_1, L_2, ... are M_{m_1}, M_{m_2}, ... and D_{x_1}, D_{x_2},..., the $(M_m)_{avg}$ and $(D_x)_{avg}$ may be approximated by

$$(M_m)_{avg} = \frac{M_{m_1} L_1 + M_{m_2} L_2 + \cdots}{L_1 + L_2 + \cdots} \tag{24}$$

$$(D_x)_{avg} = \frac{L_1 + L_2 + \cdots}{L_1/D_{x_1} + L_2/D_{x_2} + \cdots}$$

The validities of these expressions have been tested in our laboratory with B/Ep-Gr/Ep hybrid composites (B/Ep-AVCO 5505, Gr/Ep-Hercules 3501-5/A-S). Good agreement was found between the measured and calculated average values of M_m and D.

The average values $(M_m)_{avg}$ and $(D_x)_{avg}$ must be used with caution in calculating the moisture content of the material. For a period of time after the conditions in the environment have changed only the outer layer or layers will sense this change. Thus, during these early time periods, a substantial error may be introduced in the moisture content if the values $(M_m)_{avg}$ and $(D_x)_{avg}$ are used to calculate the moisture content instead of the values corresponding to the outer layer.

SECTION VI

RESULTS FOR GRAPHITE T-300 FIBERITE 1034 COMPOSITES

A series of tests was performed to measure the moisture absorption and desorption of $\pi/4$ and unidirectional Graphite T-300 Fiberite 1034 composites. The tests were conducted with the specimen a) exposed to humid air, b) exposed to saturated steam, c) fully immersed in water, and d) dried in an oven. The apparatus and the detailed results are given in Appendix B. The conditions employed in the tests are summarized in Table 1. Four unidirectional and four $\pi/4$ specimens were tested at each of the 18 test conditions. Thus, a total of 144 specimens were tested. A typical test result is shown in Figure 6.

The accuracy of the tests can be assessed by calculating G from the data (see equation 13) and by comparing the values obtained with the theoretical G values given in Figure 2. This comparison, shown in Figure 7, shows good correspondence between the data and the theory.

The maximum moisture content as a function of humidity is shown in Figure 8. A fit to the data gives

$$M_m = 0.00014 \, (\phi)^2 \qquad percent \tag{26}$$

For the material fully submerged in water the tests give

$$M_m = 1.8 \qquad percent \tag{27}$$

Equations (26) and (27) apply to both unidirectional and $\pi/4$ composites.

It is interesting to note that according to the present tests the maximum moisture content is insensitive to temperature. A slight dependence of M_m on temperature has been observed by other investigators at high temperatures. Thus the dependence of M_m on temperature merits further investigation.

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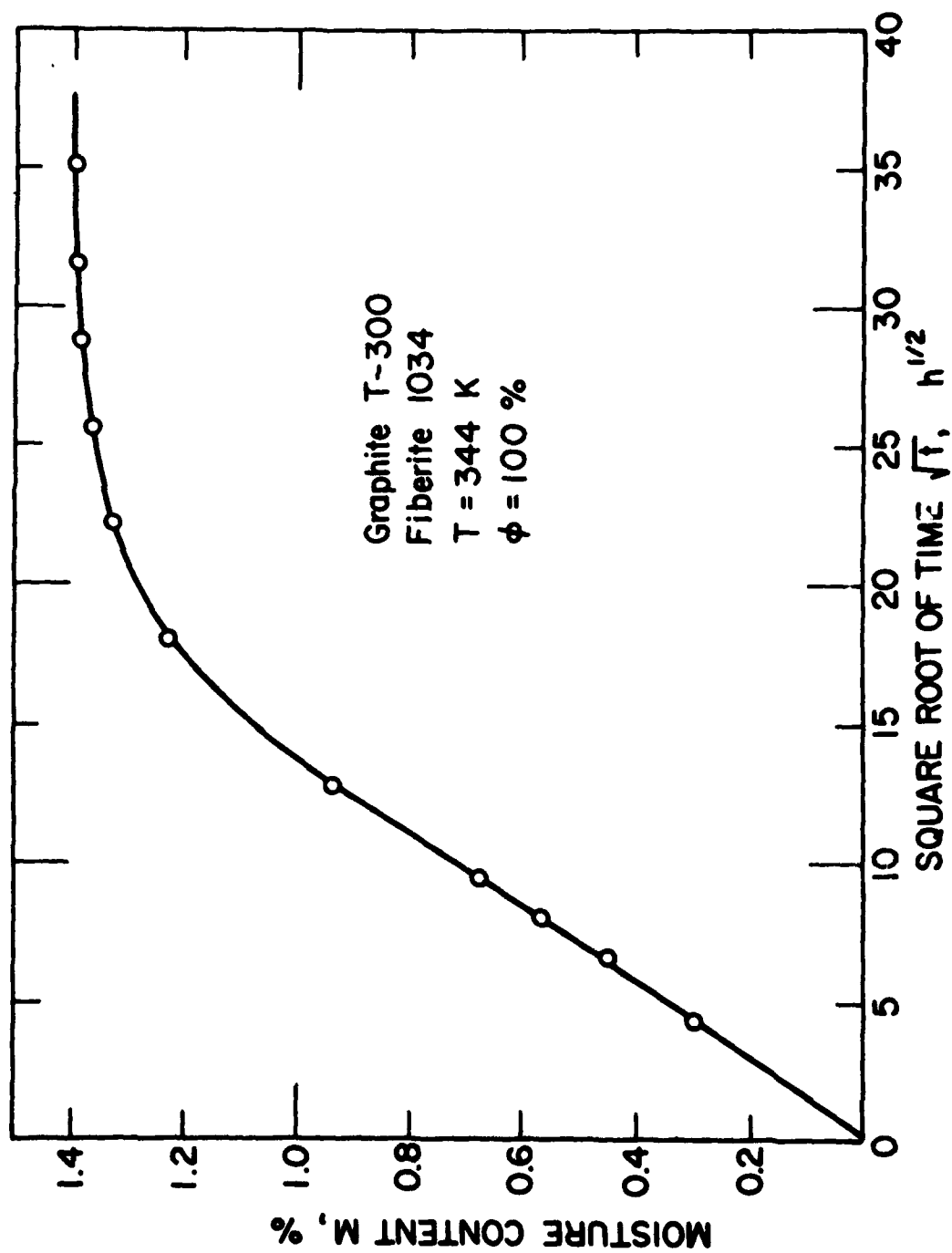
Figure 6. Typical Test Results for a Unidirectional Graphite T-300 Fiberite 1034 Composite (v_f = 0.68 and α = 90°)
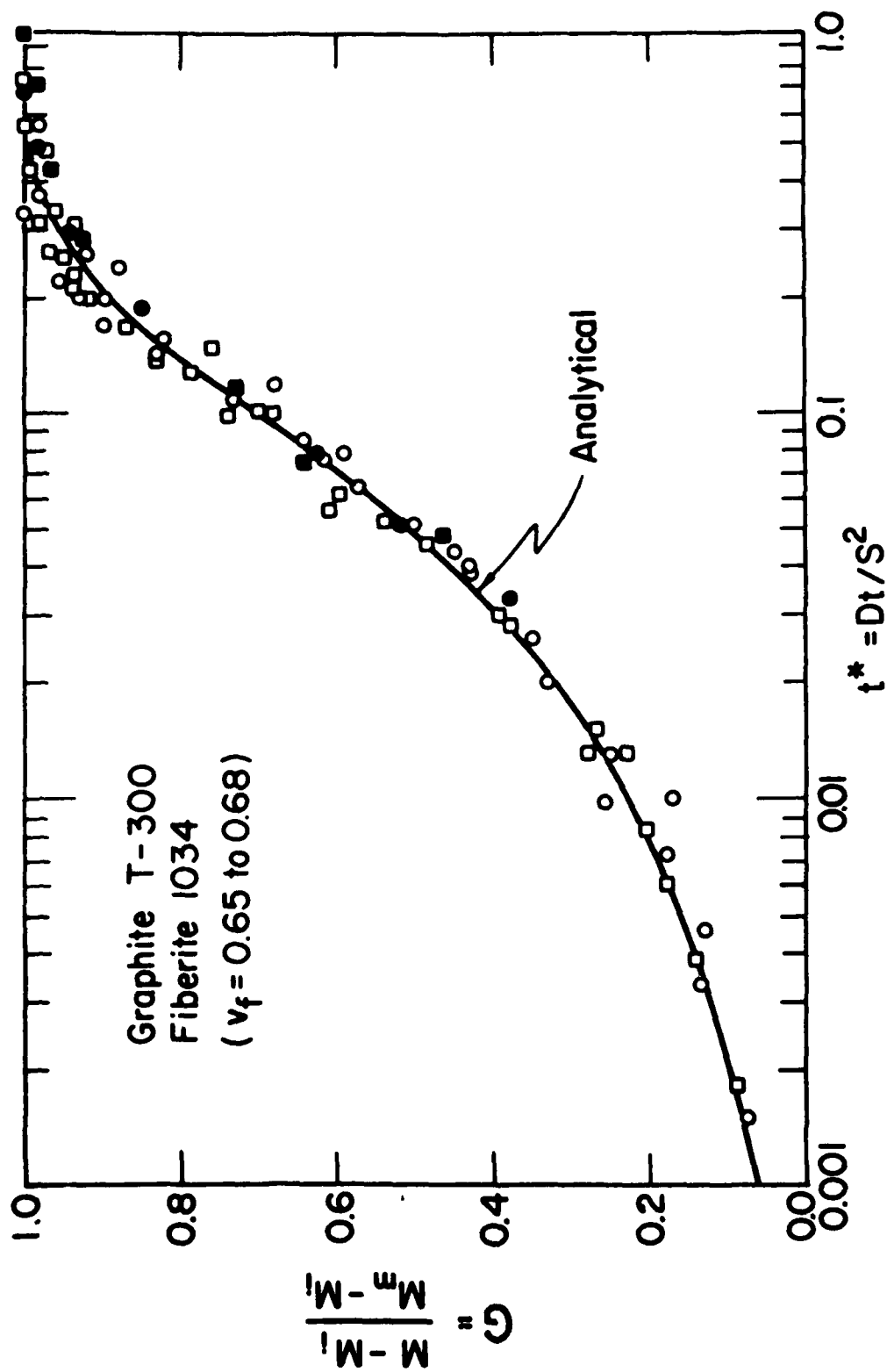
Figure 7. Comparison of Analytical (Eq. 8) and Measured G Values for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites ($v_f = 0.68$ and $\alpha = 90°$). O : Unidirectional; □ : $\pi/4$ (Empty and Dark Symbols Represent Absorption and Desorption, Respectively)
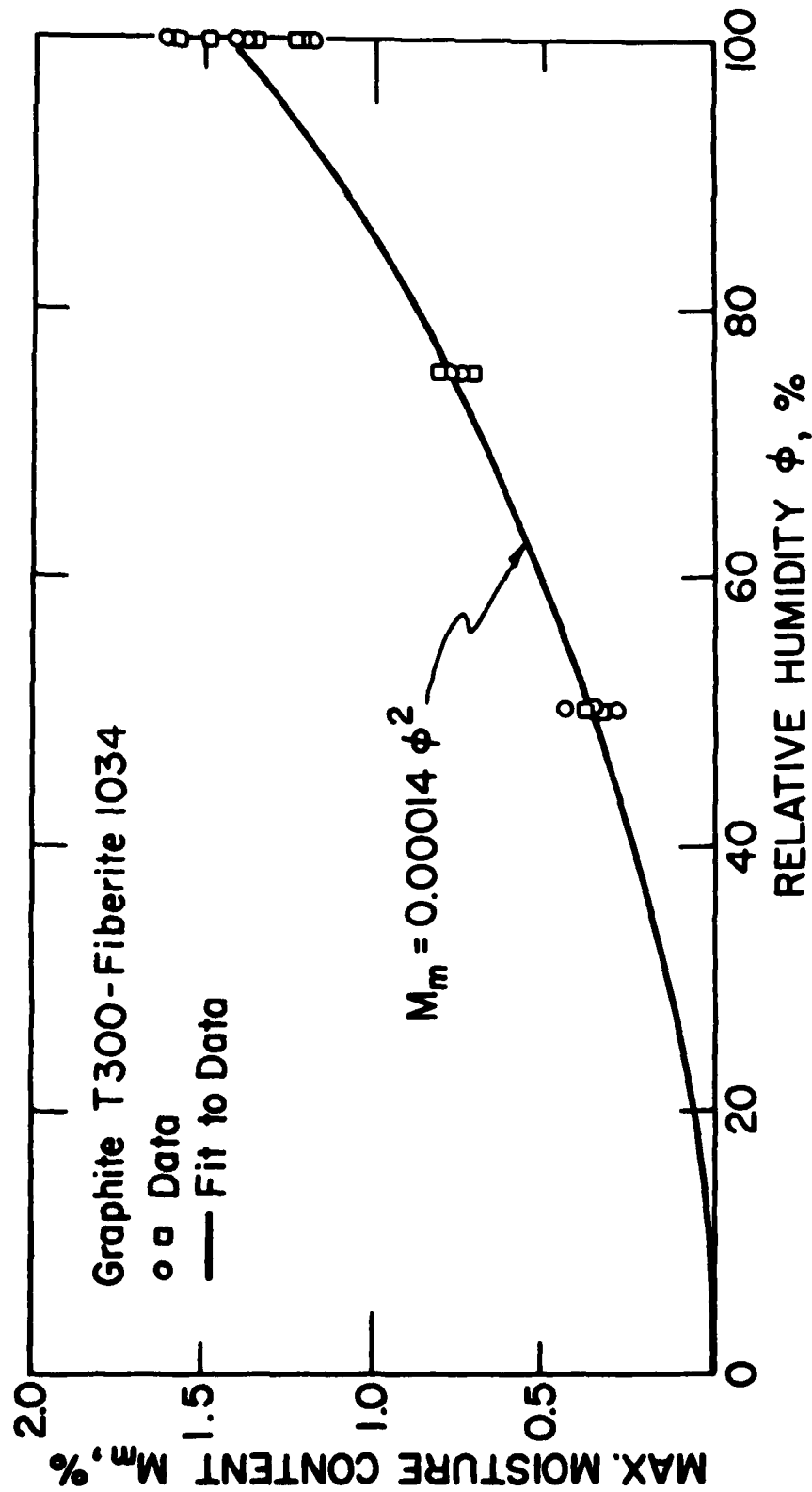
Figure 8. The Variation of the Maximum Moisture Content with Relative Humidity for Graphite T-300 Fiberite 1034 Composites. ($v_f = 0.68$ and $\alpha = 90°$) O : Unidirectional □ : $\pi/4$; —— Fit to Data

The test results for the diffusivities of the Graphite T-300 Fiberite 1034 composites were presented before in Figure 3.

It is important to note that size changes in the specimens (swelling) were also measured during the tests with a micrometer. Within the accuracy of the measurements (25.4 μm which corresponds to about 0.2% swelling) changes in specimen sizes could not be detected under any of the test conditions.

SECTION VII

SUMMARY OF RESULTS FOR SINGLE LAYERED COMPOSITES

EXPOSED TO CONSTANT ENVIRONMENTAL CONDITIONS

The following problem was investigated. A material (either homogeneous or composite) is exposed to a moist environment. Depending upon the environmental conditions and the condition of the material, the material either absorbs or loses moisture as manifested by weight gain or weight loss. The objective is to determine the percent moisture content M (percent weight gain) of the material as a function of time t

$$M = M(t) = \frac{\text{WEIGHT OF MOIST MATERIAL} - \text{WEIGHT OF DRY MATERIAL}}{\text{WEIGHT OF DRY MATERIAL}} \times 100 \qquad (11)$$

It was found that when a) the material is exposed to the environment on one side only, or on two sides with both sides being parallel (one dimensional problem, Figures 1a and 1b), b) initially the temperature and moisture distributions inside the material are uniform, and c) the moisture content and the temperature of the environment are constant, the moisture content of the material during both absorption and desorption is

$$M = G(M_m - M_i) + M_i \qquad (13)$$

M_i is the initial moisture content of the material, M_m is the maximum moisture content which can be attained under the given environmental conditions, and G is a time dependent parameter

$$G = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp[-(2j+1)^2 \pi^2 (\frac{D_z t}{s^2})]}{(2j+1)^2} \qquad (8)$$

Equation (8) may be approximated by the expression

$$G = 1 - exp\left[-7.3\left(\frac{D_x t}{s^2}\right)^{0.75}\right] \qquad (10)$$

For a material exposed on two sides to the same environment s is equal to the thickness (s=h); for a material insulated on one side (thermal insulation, impermeable to moisture) s is twice the thickness (s=2h). D_x is the diffusivity of the material in the direction normal to the surface. For fiber reinforced composites in which the orientations of all the fibers with respect to the x, y, z axes are α, β, and γ (Figure 1)

$$D_x = D_{11} \cos^2\alpha + D_{22} \sin^2\alpha \qquad (18)$$

where D_{11} and D_{22} are the diffusivities in the directions parallel and normal to the fibers (longitudinal and transverse directions). If D_x is not known it may be estimated from the diffusivity of the matrix D_r and the volume fraction of the fibers v_f

$$D_x = D_r\left[(1 - v_f)\cos^2\alpha + (1 - 2\sqrt{v_f/\pi})\sin^2\alpha\right] \qquad (19)$$

Equation (19) may be applied to unidirectional and laminated composites (e.g. $\pi/4$ composites). In the latter case in each laminate a) D_r must be the same, b) the volume fraction of the fibers v_f must be the same, and c) the fibers must be in a plane parallel to the surface ($\alpha = 90°$). The orientations of the fibers relative to the y, z directions may vary from laminate to laminate.

Experimental evidence indicates that the maximum moisture content is insensitive to the temperature but depends on the moisture content of the environment. For a material immersed in liquid M_m is a constant

$$M_m = \text{CONSTANT} \qquad \text{(fully immersed)} \tag{28}$$

For a material exposed to humid air M_m depends on the relative humidity according to the relationship

$$M_m = a \phi^b \qquad \text{(humid air)} \tag{20}$$

The time required for a material to attain at least 99.9 percent of its maximum possible moisture content is

$$t_m = \frac{0.67 S^2}{D_x} \tag{9}$$

The time required to reach the maximum moisture content is insensitive to the moisture content of the environment but depends on the temperature through D_x.

The foregoing results for M and t_m are based on an exact solution of the equations governing the problem (Section III). However, in order to calculate M and t_m the maximum moisture content M_m (or the constants a and b) and the diffusivity D_x must be known. These parameters must be determined experimentally. The accuracy of M and t_m depend directly on the accuracy of the experiments. Proper test procedures for determining M_m, a, b, and D_x are described in Section V.

An extensive series of tests were performed studying the moisture absorption and desorption characteristics of Graphite T-300 Fiberite 1034 composites. These tests gave the following results:

Graphite T-300 Fiberite 1034 exposed to humid air

$$M_m = 0.00014(\phi)^2 \qquad percent \qquad (26)$$

Graphite T-300 Fiberite 1034 immersed in water

$$M_m = 1.8 \qquad percent \qquad (27)$$

The matrix diffusivity together with the longitudinal and transverse diffusivities are shown in Figure 3.

SECTION VIII

TIME VARYING BOUNDARY CONDITIONS

The moisture content given by equation (8) is applicable only for single layered composites when the initial temperature and moisture concentration inside the material are uniform, and the temperature and moisture content of the environment are constant. When these conditions are not met and when, in addition, the conditions on the two sides of the plates differ the solution to the problem must be obtained by numerical means. The general one dimensional problem is illustrated in Figure 9. This problem is specified by the following parameters

1) thickness of each layer L_1, L_2, ...

2) thermal conductivity of each layer K_{x1}, K_{x2}, ...

3) diffusivity as a function of the temperature of each layer D_{x_1}, D_{x_2}...

4) maximum moisture content as a function of the humidity of each layer M_{m_1}, M_{m_2}, ...

5) temperature on the two exposed sides of the material as a function of time

6) relative humidity on the two exposed sides of the material as a function of time.

A computer code for the general problem described above was developed at The University of Michigan and may be obtained from the Fluid Dynamics Laboratory, Department of Mechanical Engineering, The University of Michigan. This program computes the moisture distribution in each layer and the moisture content (weight gain) of each layer as a function of time.
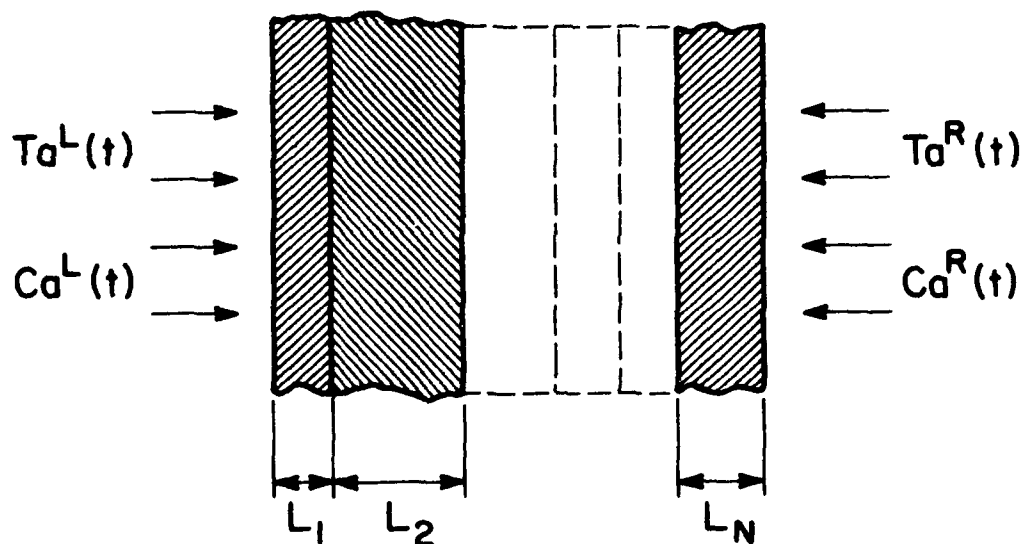
Figure 9. Model of Multilayered Material Used in the Computer Solution

SECTION IX

NON-DESTRUCTIVE TESTS FOR DETERMINING THE MOISTURE CONTENT

In the laboratory the moisture content of the material is determined by weighing the specimens. In the field this procedure is not feasible. It would be desirable therefore, to develop non-destructive test procedures which indicate the moisture content of the material. Two different types of tests were evaluated during the course of this work. First, an attempt was made to correlate the moisture content with changes in the electric resistance of the material. Second, a correlation was sought between the moisture content and the hardness of the material.

(1) Electric Resistance Measurements

The electric resistance of graphite T-300 Fiberite 1034 composites was measured on specimens 13 mm x 13 mm x 1.3 mm (8-ply). Both unidirectional and $\pi/4$ specimens were tested with the graphite fibers embedded parallel to the two largest surfaces. In order to measure the resistance between these surfaces, 6 mm thick and 12 mm diameter aluminum electrodes were placed on the surfaces directly opposite to each other. The aluminum electrodes were held in position by a Rima Spring Tester. With this tester, steady pressures of up to 15 MPa could be applied to the specimen.

The aluminum electrodes were connected to a Simpson Meter and the resistance of the specimen was read directly from the Simpson Meter. In the first series of measurements only thoroughly dried specimens were tested so as to establish the baseline values. The electric resistance was recorded as a function of the pressure applied to the specimen (pressure range: 2.7-8.3 MPa). It was found that the resistance measurements varied randomly with pressure, ranging from 0.25 to 4 kΩ. Furthermore, these values could

not be repeated. It was thought that maybe these values were not taken at
steady state. A new series of tests was undertaken to investigate the steadi-
ness of the electric resistance as a function of time. At a given constant
pressure (3.4 and 5.2 MPa) the resistance was measured at 10 minute inter-
vals up to one hour. The resistance values were observed to vary randomly
ranging from 0.05 to 4 kΩ. Both unidirectional and $\pi/4$ specimens showed this
random behavior. Therefore, the electric resistance measurements were not
pursued further.

(2) Surface Hardness Measurements

The surface hardness of 102 mm x 12.7 mm x 1.3 mm (8-ply) Graphite T-3C0
Fiberite 1034 composites was measured. Both unidirectional and $\pi/4$ speci-
mens were tested. The measurements were performed with a Rockwell Hardness
Tester using a 3.175 mm diameter indenter. Each specimen was thoroughly dried
and its surface hardness determined to establish the baseline hardness value.
The specimens were then put into the steam chamber (Appendix B) at 394 K.
After the desired amount of moisture was absorbed by a specimen, the sur-
face hardness of the moist specimen was measured and compared to its own dry
baseline value. The unidirectional specimens, including both dry and moist
ones, tended to crack during the hardness measurements. Thus only the mea-
surements with the $\pi/4$ specimens are reported. A plot of the percent reduc-
tion in surface hardness versus the percent of moisture content in the $\pi/4$
specimens is given in Figure 10. Although the individual points scatter
over a wide range, the mean of several measurements show a clear relation-
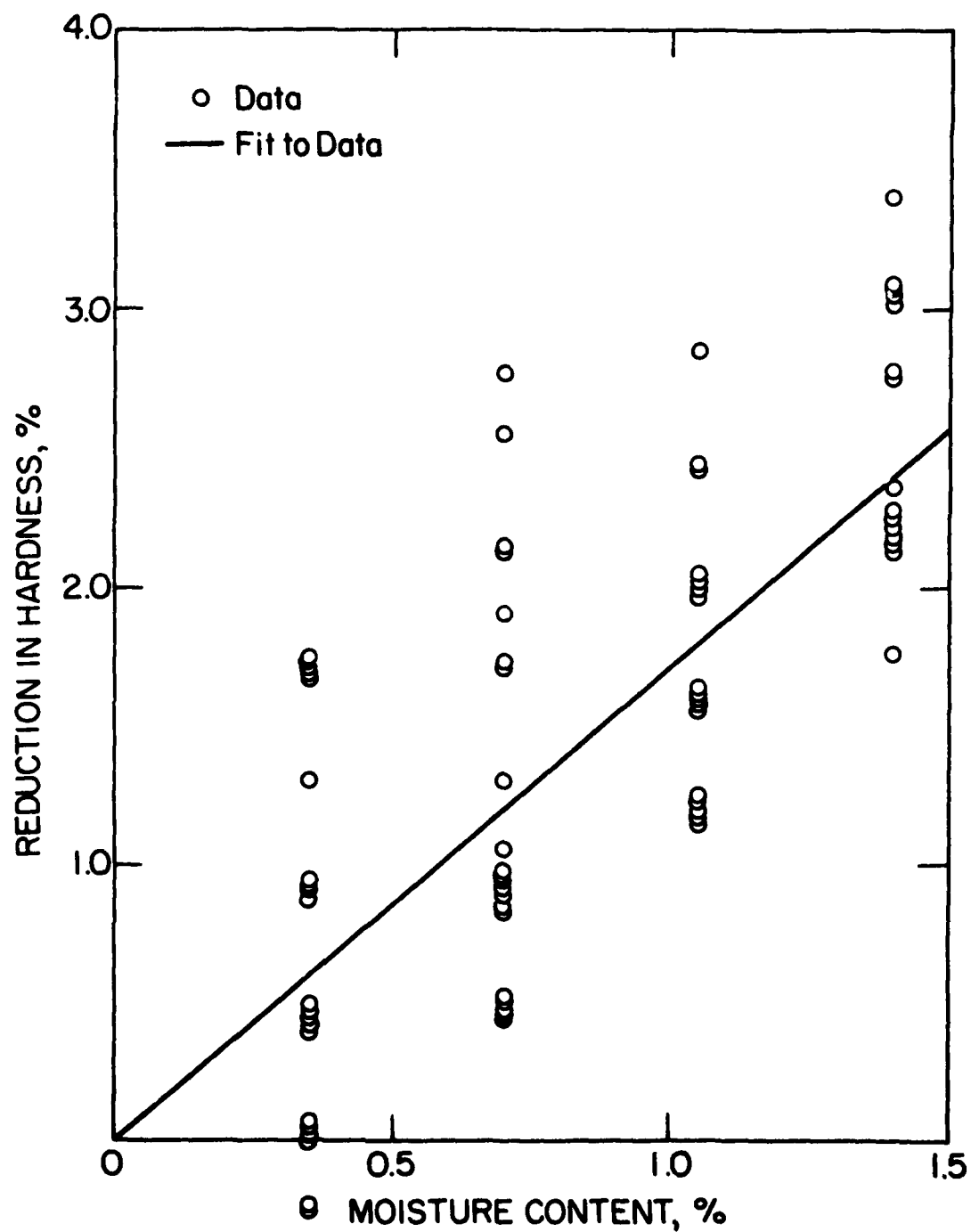ship between the moisture content and the hardness.

Figure 10.   The Relationship Between Surface Hardness and Moisture
Content of Graphite T-300 Fiberite 1034 Composite

APPENDICES A & B

CORRECTIONS FOR EDGE EFFECTS

To determine the diffusivity D_x we refer to equation (4)

$$\frac{\partial c}{\partial t} = D_x \frac{\partial^2 c}{\partial x^2}$$

For a semi-infinite solid ($h \to \infty$) the boundary conditions (equations 5a and 5b) become

$$c = c_i \qquad\qquad 0 < x < \infty \qquad t \le 0 \qquad\qquad \text{(A1a)}$$

$$c = c_m \qquad\qquad x = 0 \qquad t > 0 \qquad\qquad \text{(A1b)}$$

In this case the concentration as a function of position and time in the solid is (Reference 3)

$$\frac{c - c_i}{c_m - c_i} = 1 - erf \frac{x}{2\sqrt{D_x t}} \qquad\qquad \text{(A2)}$$

and the weight of moisture which enters or leaves through an area A in time t is

$$m = \int_0^t -A D_x \left(\frac{\partial c}{\partial x}\right)_{x=0} g \, dt \qquad\qquad \text{(A3)}$$

Equations (A2) and (A3) give (Reference 3)

$$m = 2 A g (c_m - c_i) \sqrt{\frac{D_x t}{\pi}} \qquad\qquad \text{(A4)}$$

During a test moisture enters or leaves the specimen on all six sides (Figure 4). In the early stages of the process the interaction of the different sides may be neglected and equation (A4) can be applied to each side independently. Thus the total weight of the entering moisture is

$$m = 4g(C_m - C_i)(nl\sqrt{D_x} + nh\sqrt{D_y} + hl\sqrt{D_z})\sqrt{\frac{t}{\pi}} \qquad (A5)$$

where D_x, D_y, and D_z are the diffusivities in the x, y, and z directions. For an initially dry specimen $[c_i = 0$ and $W_d = \rho g h n l]$ the moisture content as a function of time is

$$M = \frac{W - W_d}{W_d} \times 100 = \frac{m}{W_d} \times 100 = \frac{4C_m}{\rho h}\left(\sqrt{D_x} + \frac{h}{l}\sqrt{D_y} + \frac{h}{n}\sqrt{D_z}\right)\sqrt{\frac{t}{\pi}} \qquad (A6)$$

Note that C_m/ρ is the maximum moisture content

$$M_m = \frac{W_m - W_d}{W_d} \times 100 = \frac{m_m/(h n l)}{W_d/(h n l)} \times 100 = \frac{C_m}{\rho} \qquad (A7)$$

Hence

$$M = \frac{4 M_m}{h\sqrt{\pi}}\left(\sqrt{D_x} + \frac{h}{l}\sqrt{D_y} + \frac{h}{n}\sqrt{D_z}\right)\sqrt{t} \qquad (A8)$$

Equation (A8) shows that M varies linearly with $\sqrt{t}$ at least for a short time after exposure to the environment, i.e. before the opposing sides begin to affect significantly the moisture content. Equation (A8) may be written as

$$M = \frac{4 M_m}{h}\sqrt{\frac{t}{\pi}}\sqrt{D} \qquad (A9)$$

where

$$D = D_x\left(1 + \frac{h}{l}\sqrt{\frac{D_y}{D_x}} + \frac{h}{n}\sqrt{\frac{D_z}{D_x}}\right)^2 \qquad (A10)$$

For a homogeneous material $D_x = D_y = D_z$ and equation (A10) becomes

$$D = D_x\left(1 + \frac{h}{l} + \frac{h}{n}\right)^2 \qquad (A11)$$

For composites the diffusivities D_x, D_y, and D_z must first be evaluated.

For unidirectional composites (fiber orientations α, β, and γ, Figure 1), the diffusivities are (Reference 5)

$$D_x = D_{11} \cos^2\alpha + D_{22} \sin^2\alpha \tag{A12a}$$

$$D_y = D_{11} \cos^2\beta + D_{22} \sin^2\beta \tag{A12b}$$

$$D_z = D_{11} \cos^2\gamma + D_{22} \sin^2\gamma \tag{A12c}$$

Equations (17) and (A12) together with equation (A10) give D for unidirectional composites

$$D = D_r\left[(1-V_f)\cos^2\alpha + (1-2\sqrt{V_f/\pi})\sin^2\alpha\right]\left[1 + \frac{h}{\ell}\sqrt{\frac{(1-V_f)\cos^2\beta + (1-2\sqrt{V_f/\pi})\sin^2\beta}{(1-V_f)\cos^2\alpha + (1-2\sqrt{V_f/\pi})\sin^2\alpha}} \right.$$
$$\left. + \frac{h}{n}\sqrt{\frac{(1-V_f)\cos^2\gamma + (1-2\sqrt{V_f/\pi})\sin^2\gamma}{(1-V_f)\cos^2\alpha + (1-2\sqrt{V_f/\pi})\sin^2\alpha}}\right]^2 \tag{A13}$$

We consider next laminated composites consisting of N layers. The thickness of the jth layer is h_j. In the jth layer the fiber orientations with respect to the y and z axes are β_j and γ_j. The fiber orientation with respect to the x axis is 90° in every laminate, and $\gamma_j = 90° - \beta_j$. For these conditions equation (A12) gives

$$D_x = D_{22} \tag{A14a}$$

$$D_y = D_{11} \frac{\sum_{j=1}^{N} h_j \cos^2\beta_j}{\sum_{j=1}^{N} h_j} + D_{22} \frac{\sum_{j=1}^{N} h_j \sin^2\beta_j}{\sum_{j=1}^{N} h_j} \tag{A14b}$$

$$D_z = D_{11} \frac{\sum_{j=1}^{N} h_j \sin^2\beta_j}{\sum_{j=1}^{N} h_j} + D_{22} \frac{\sum_{j=1}^{N} h_j \cos^2\beta_j}{\sum_{j=1}^{N} h_j} \tag{A14c}$$

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Combining equations (17), (A10), and (A14), we obtain an expression for D for laminated composites

$$D = D_r(1-2\sqrt{U_f/\pi})\left[1 + \frac{\lambda}{\ell}\sqrt{\frac{(1-U_f)\sum\limits_{j=1}^{N}\lambda_j\cos^2\beta_j}{(1-2\sqrt{U_f/\pi})\sum\limits_{j=1}^{\tilde{N}}\lambda_j}} + \frac{\sum\limits_{j=1}^{N}\lambda_j\sin^2\beta_j}{\sum\limits_{j=1}^{\tilde{N}}\lambda_j}\right.$$

$$\left. + \frac{\lambda}{n}\sqrt{\frac{(1-U_f)\sum\limits_{j=1}^{\tilde{N}}\lambda_j\sin^2\beta_j}{(1-2\sqrt{U_f/\pi})\sum\limits_{j=1}^{\tilde{N}}\lambda_j}} + \frac{\sum\limits_{j=1}^{N}\lambda_j\cos^2\beta_j}{\sum\limits_{j=1}^{N}\lambda_j}\right]^2 \qquad (A15)$$

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APPENDIX B

DESCRIPTION OF APPARATUS AND DATA SUMMARY

(1) Description of Apparatus

The moisture absorption and desorption characteristics of the graphite
T-300 Fiberite 1034 composites were determined by placing the composite
specimens inside environmental chambers in which the temperature and the
moisture content could be regulated. A total of twelve chambers were used
simultaneously. Each chamber was made of a 0.1 m^3 galvanized iron tank. The
required temperature inside each environmental chamber was maintained by an
electric heater placed at the bottom of the chamber. Power from the building
supply line was fed to the electric heaters through individual voltage regu-
lators. Fiberglas insulations were wrapped around the outside of the cham-
ber to minimize heat losses. The temperature inside the chamber was moni-
tored by a thermocouple placed next to the specimens.

During desorption tests (relative humidity = 0%), chemical drying agents
(1 kg of anhydrous calcium chloride and 0.1 kg of anhydrous calcium sulfate)
were placed inside the chamber to absorb the water vapor from the air. During
absorption tests in constant humidity environments (50% and 75% humidity), two
continuous streams of dry air were fed into the chamber (Figure B·1). The in-
let air pressures were slightly above atmospheric ($\sim$ 0.5 k Pa). One of the
air streams was connected to a 4 ℓ glass bottle half filled with water. The
air was bubbled through the water and became saturated with water vapor at
the chamber temperature. This moist air was next fed into a 1 ℓ glass bottle
which served as a trap for the water droplets dispersed in the moist air.
The other air stream was fed directly into the chamber where it was mixed
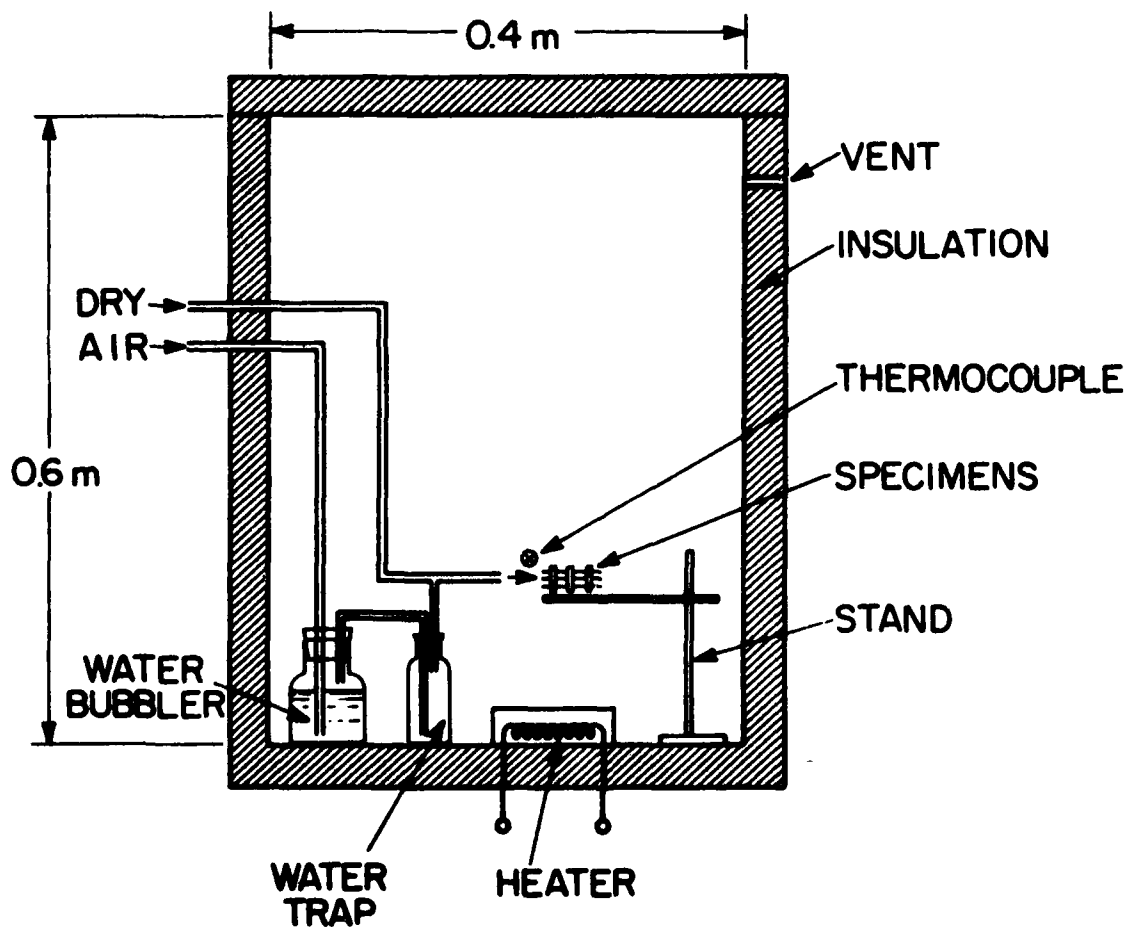with the saturated air stream. The mixture of moist and dry air stream was

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Figure B·1.   Schematic of Apparatus

directed towards the specimens. The air left through a 10 mm hole near the top of the chamber. The necessary humidity was attained inside the chamber by adjusting the flowrates of the two air streams. In order to expose the specimens to 100% humidity, the chamber was partially filled with water and heated to the required temperature. The specimens were then placed right above the water surface where the humidity was taken to be 100%. In these tests the air was stagnant; there was no air flowing through the chamber. To determine the absorption characteristic of the material during immersion in water specimens were also immersed in the water maintained at the desired temperature.

A steam chamber was constructed to test specimens under saturated steam conditions. This chamber was made of mild steel and had an inside dimension of 90 mm x 230 mm x 300 mm. Steam from the supply line in the building was fed into the chamber continuously during the tests. The conditions of the steam inside the chamber were measured by a pressure gauge and a hot-well thermometer. The desired steam condition was attained by regulating the flowrate of the steam through the chamber.

(2) Data Summary

Both the unidirectional and the $\pi/4$ specimens tested were 102 mm long, 12.7 mm wide, and 8 plus thick (~ 1.3 mm). The test conditions are listed in Table 1. The results of the tests are presented in Figures B·2-B·19.

Table 1. Summary of Conditions Used in the Tests with Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites. For All Specimens Fiber Volume Fraction v_f = 0.65 to 0.68; Fiber Orientation α = 90°

Moisture content	Temperature K						Remarks
Humid air 100%	300,	322,	344,	366			Absorption
75%	300,	322,	344,	366			Absorption
50%	-	322,	344,	366			Absorption
0%	-	-	-	366,	-	422	Desorption
Saturated steam	-	-	-	-	394,	422	Absorption
Immersed in water	-	-	344,	366,	-	422	Absorption
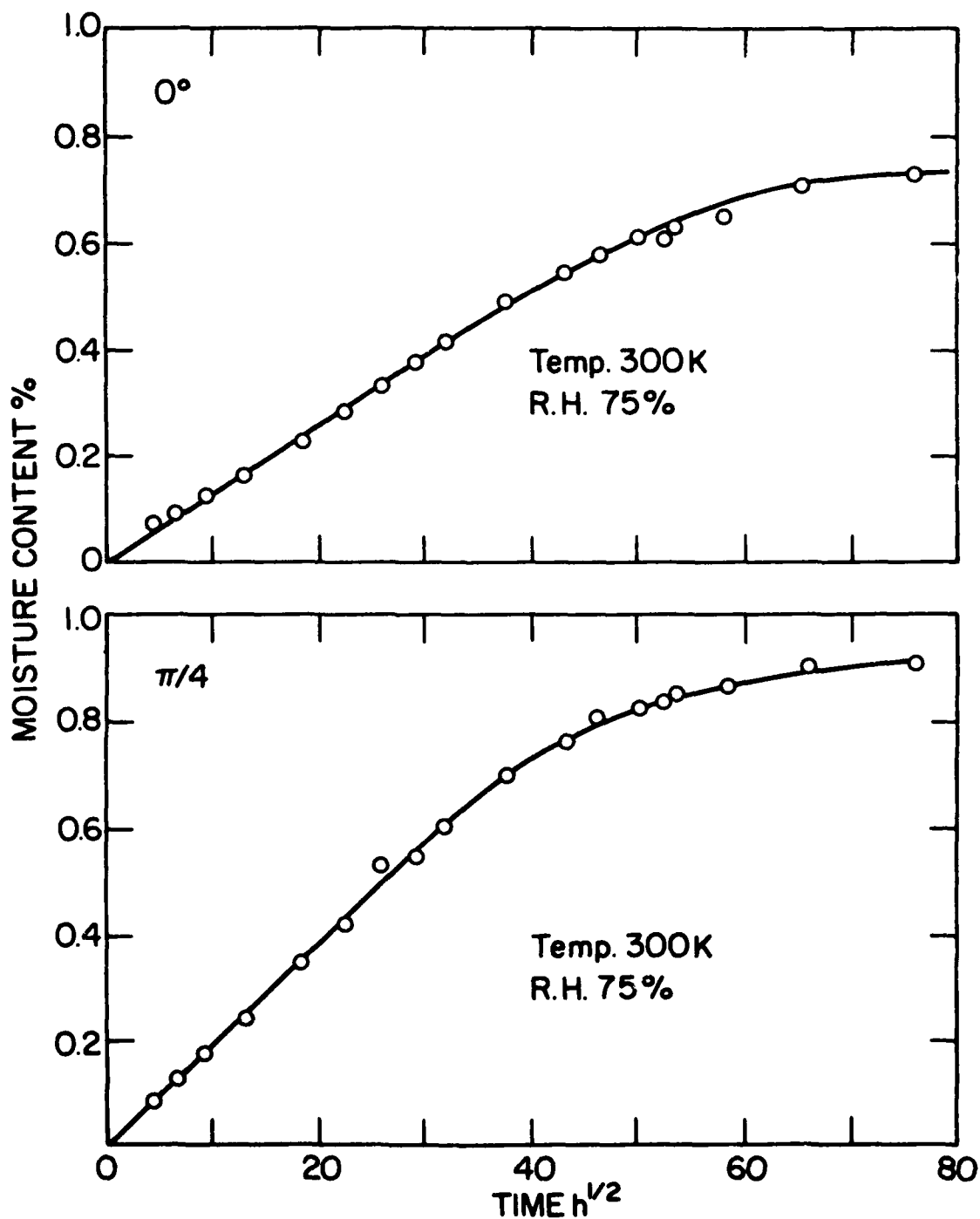
Figure B·2. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 75% Relative Humidity at 300 K. Circle Represents the Average of Four Data Points with the Diameter of each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
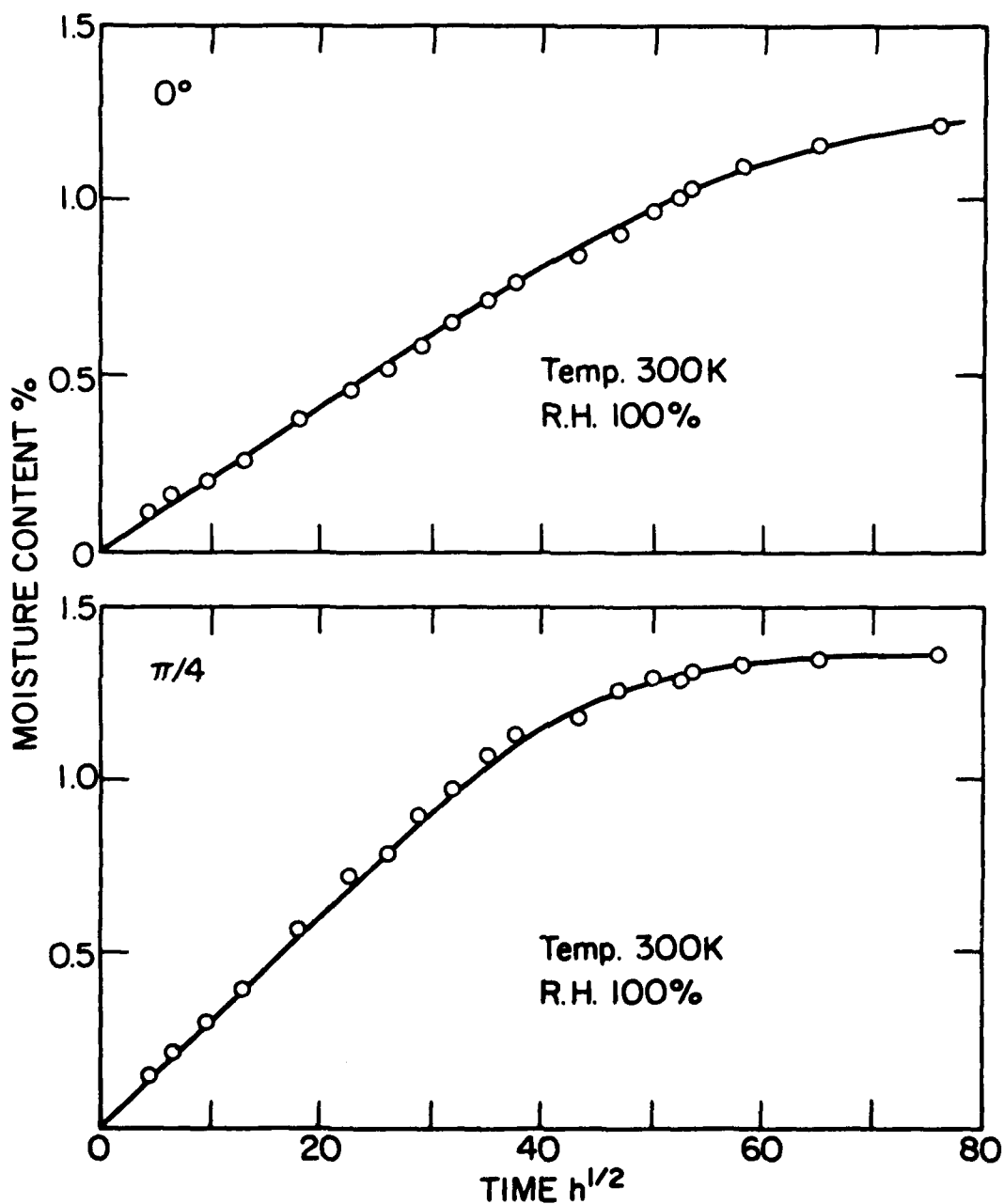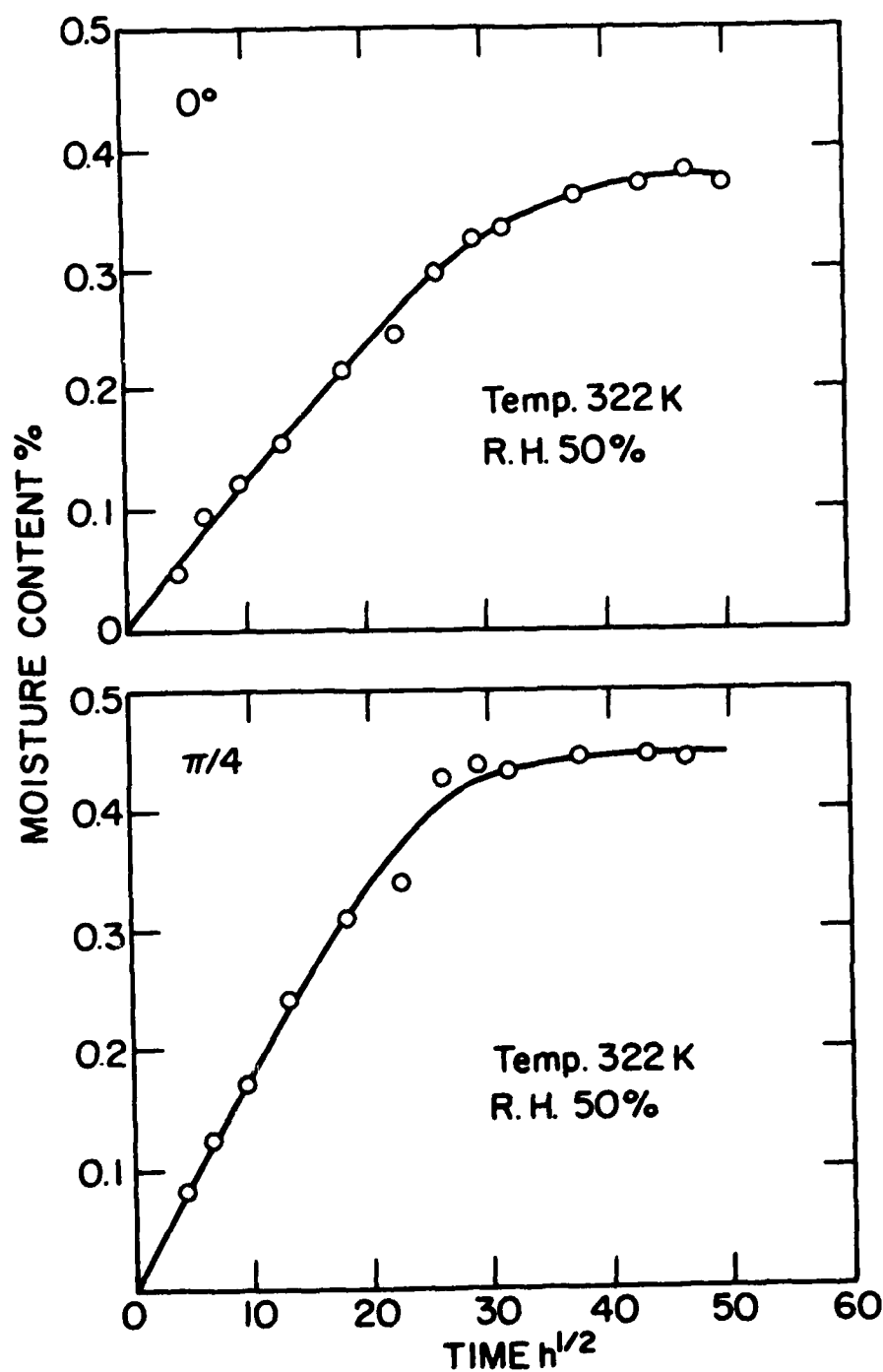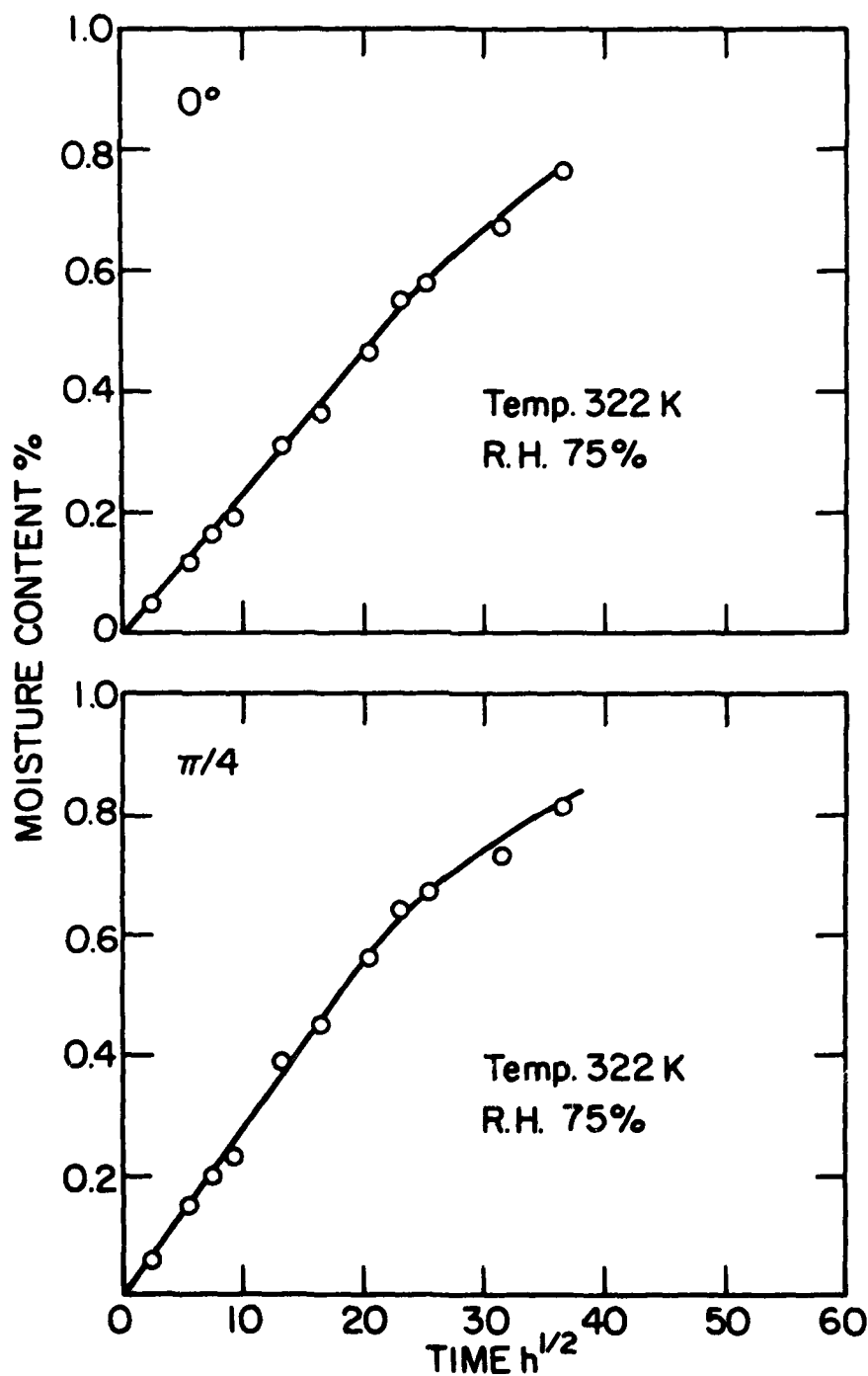
Figure B·3. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 100% Relative Humidity at 300 K. Circle Represents the Average of Four Data Points with the Diameter of each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
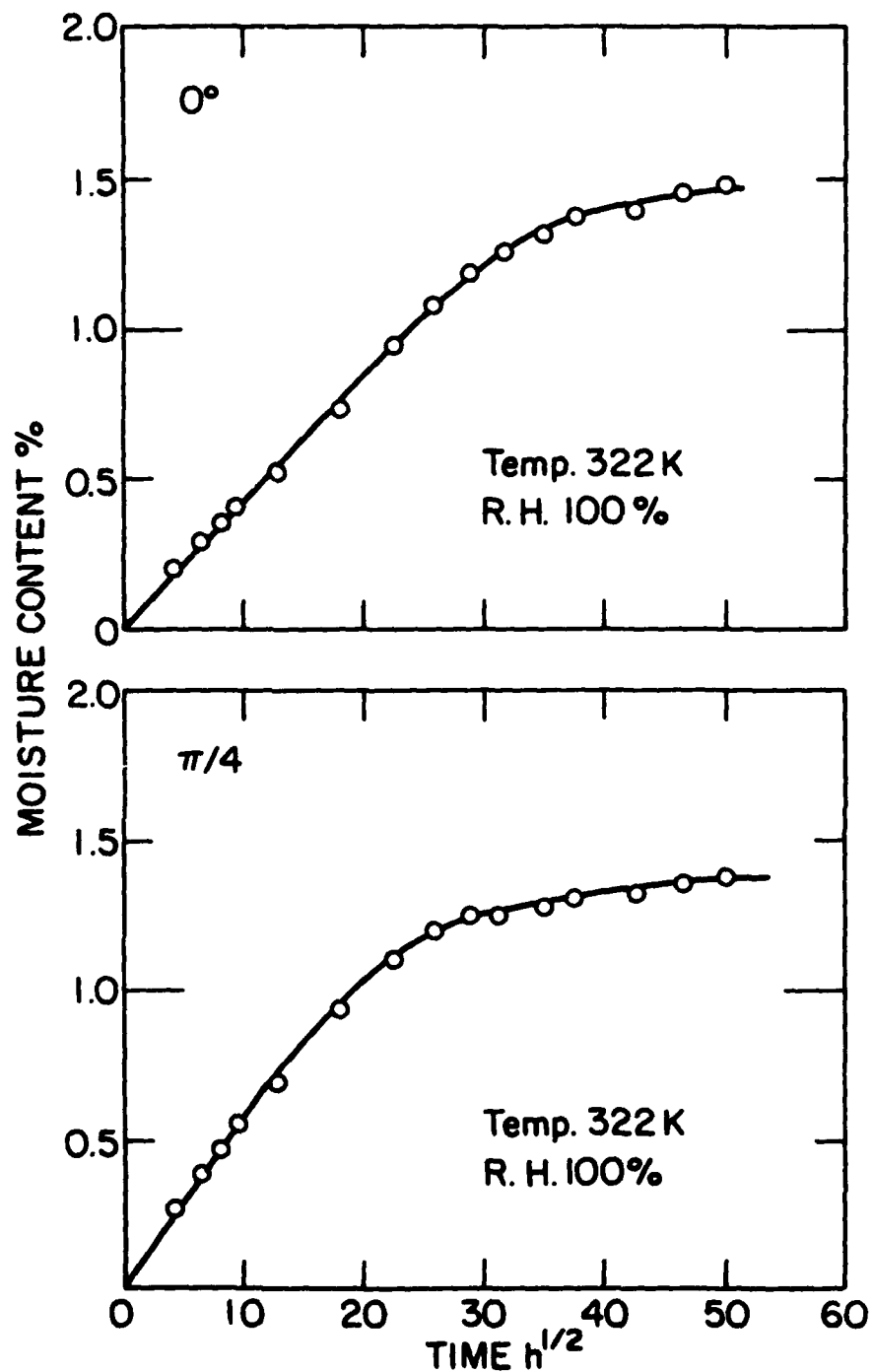
Figure B·4. Moisture Content Versus Exposure Time for Unidirectional and
π/4 Graphite T-300 Fiberite 1034 Composites Exposed to 50%
Relative Humidity at 322 K. Circle Represents the Average of
Four Data Points with the Diameter of each Circle Representing
the Spread in the Data. Solid Line is Fit to Data.

Figure B·5. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 75% Relative Humidity at 322 K. Circle Represents the Average of Four Data Points with the Diameter of each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
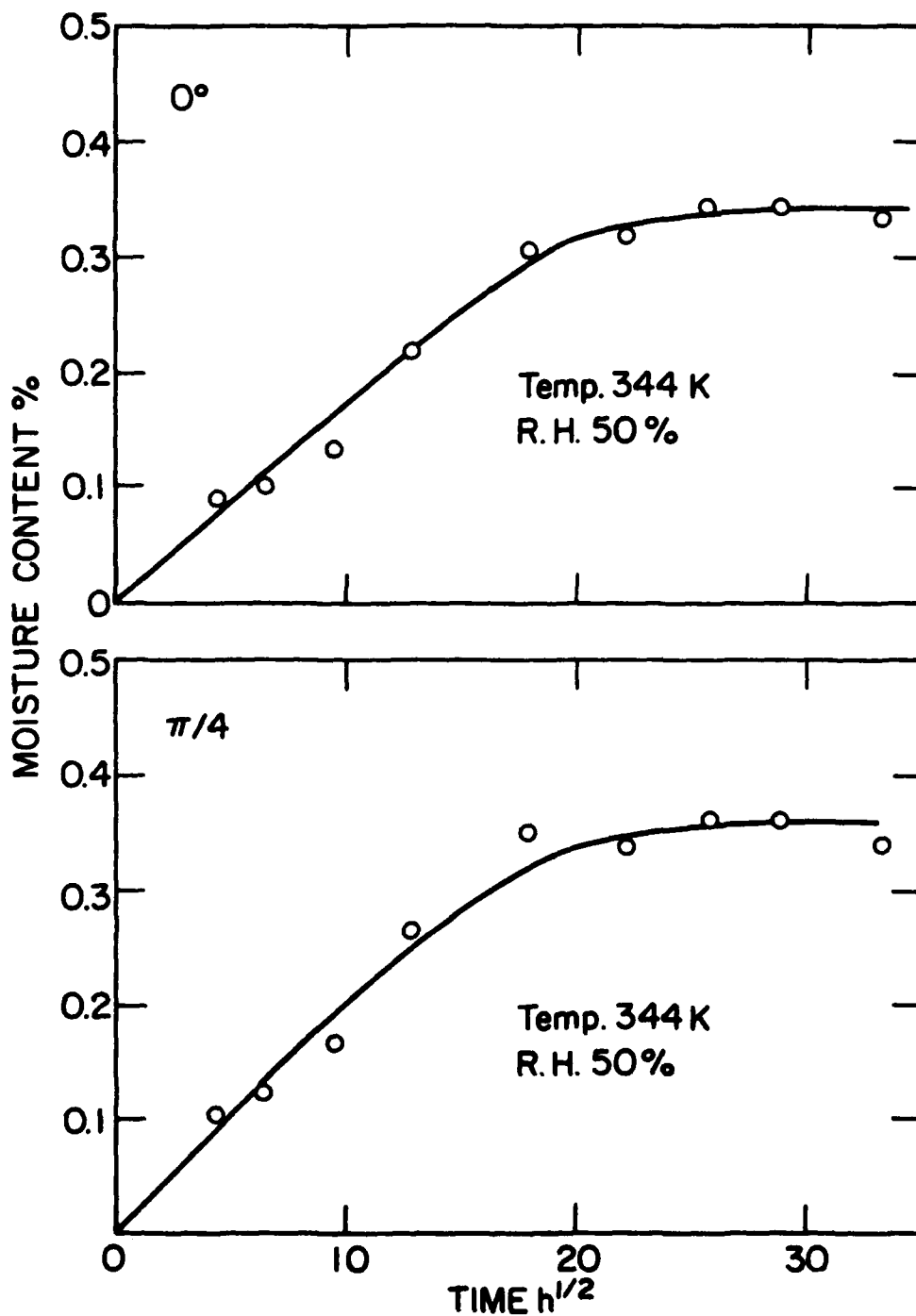
Figure B·6. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 100% Relative Humidity at 322 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
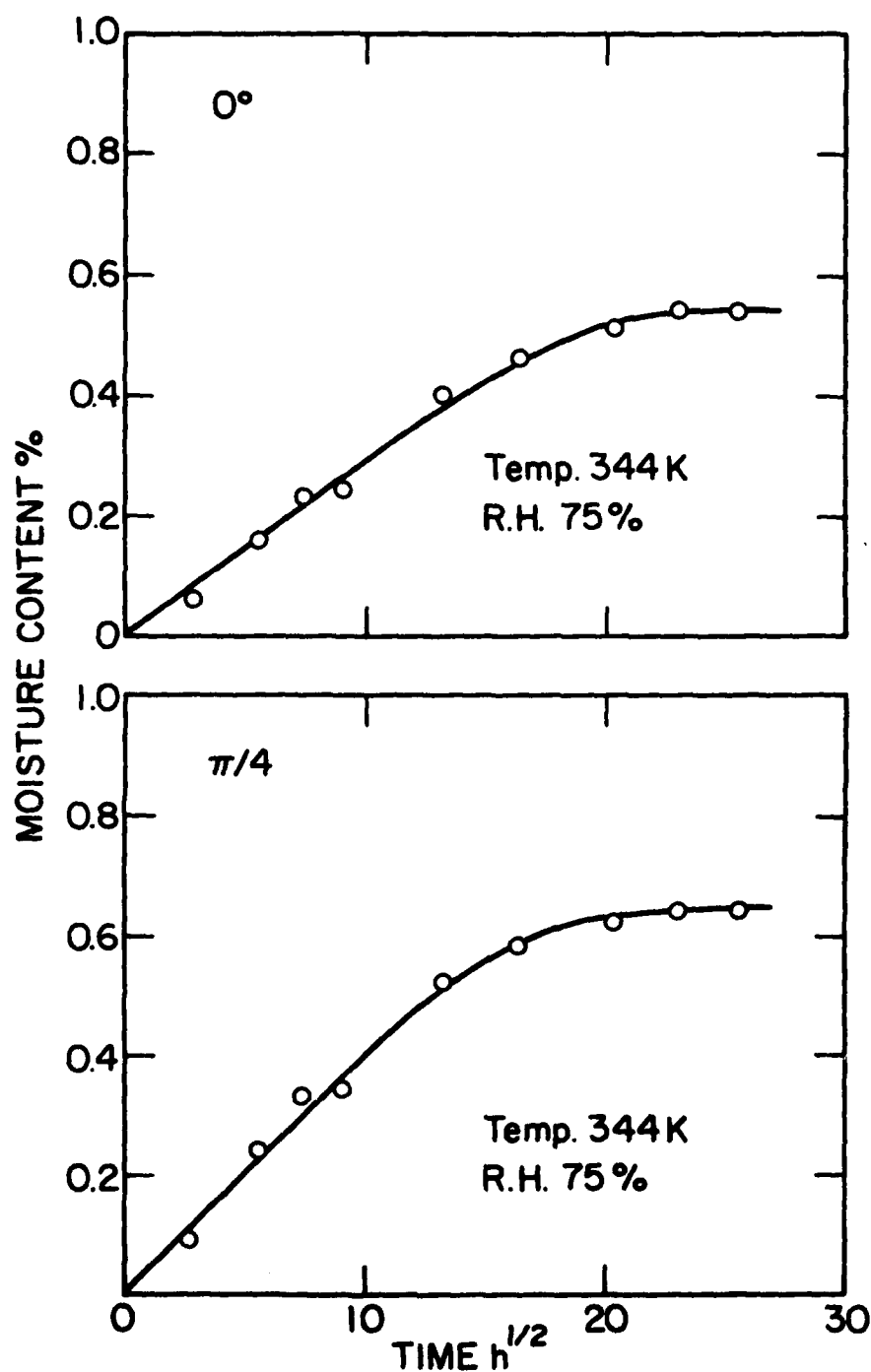
Figure B·7. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed
to 50% Relative Humidity at 344 K. Circle Represents the
Average of Four Data Points with the Diameter of Each
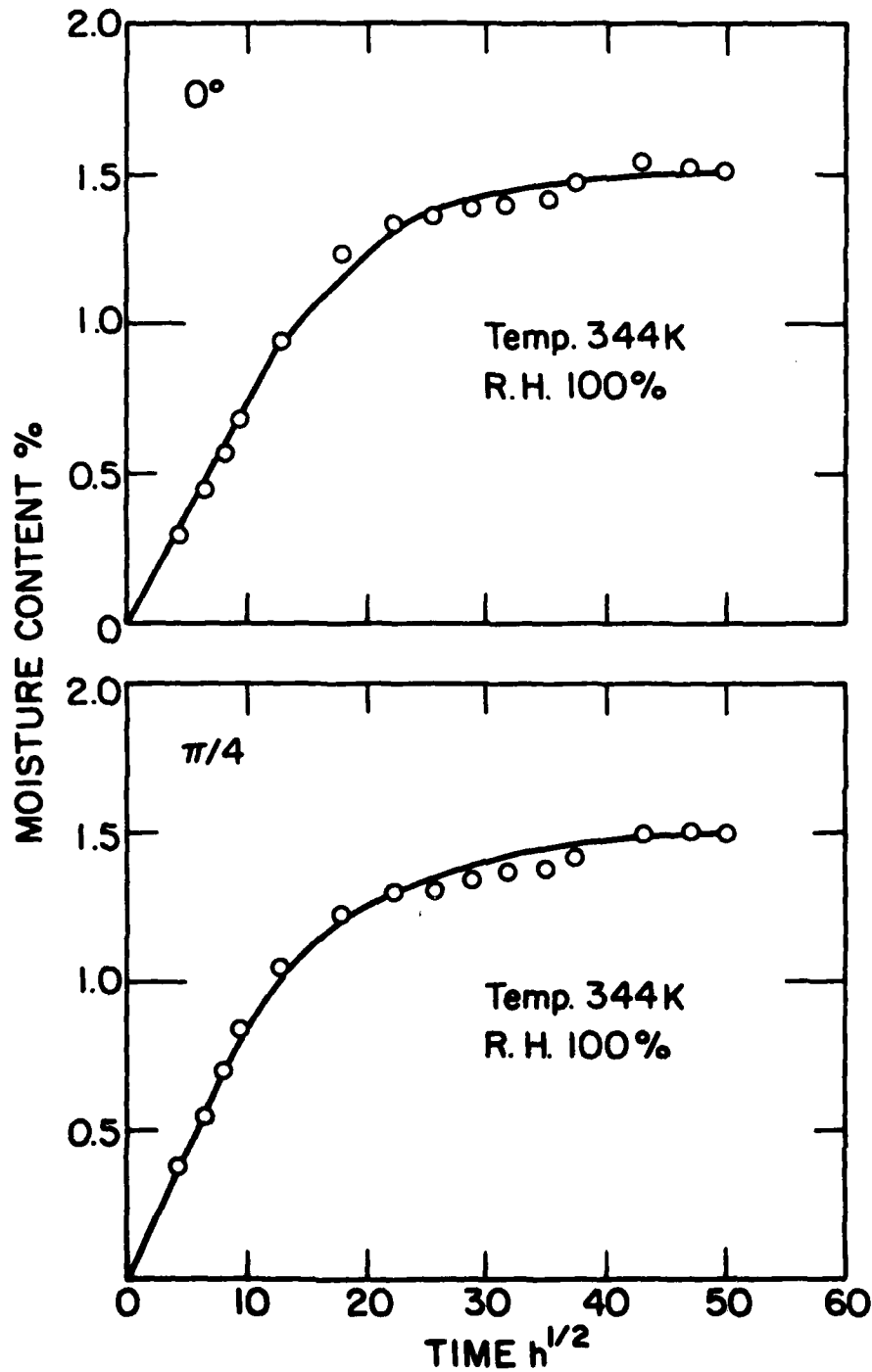Circle Representing the Spread in Data. Solid Line is
Fit to Data.

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Figure B·8. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 75% Relative Humidity at 344 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
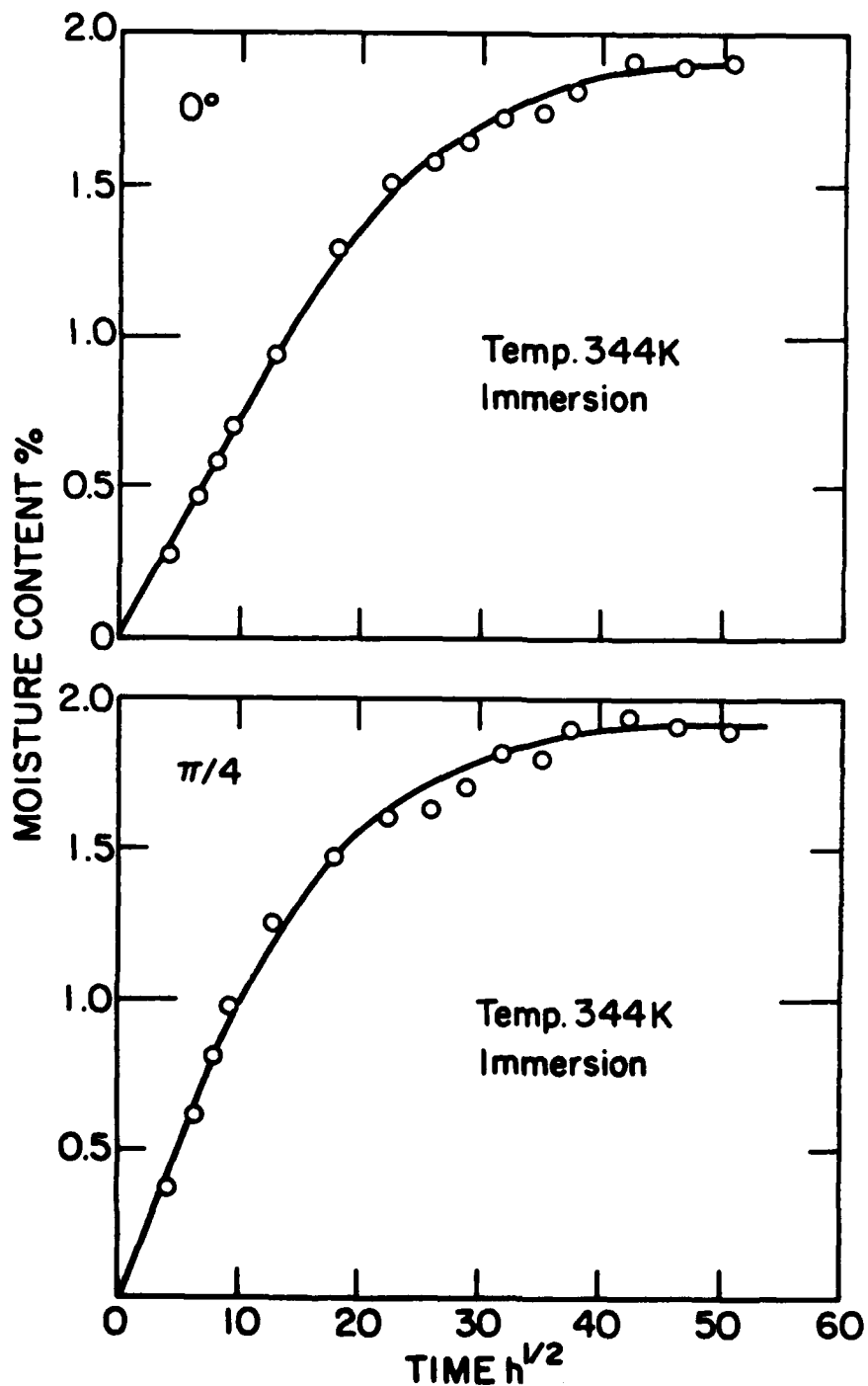
Figure B·9. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 100% Relative Humidity at 344 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
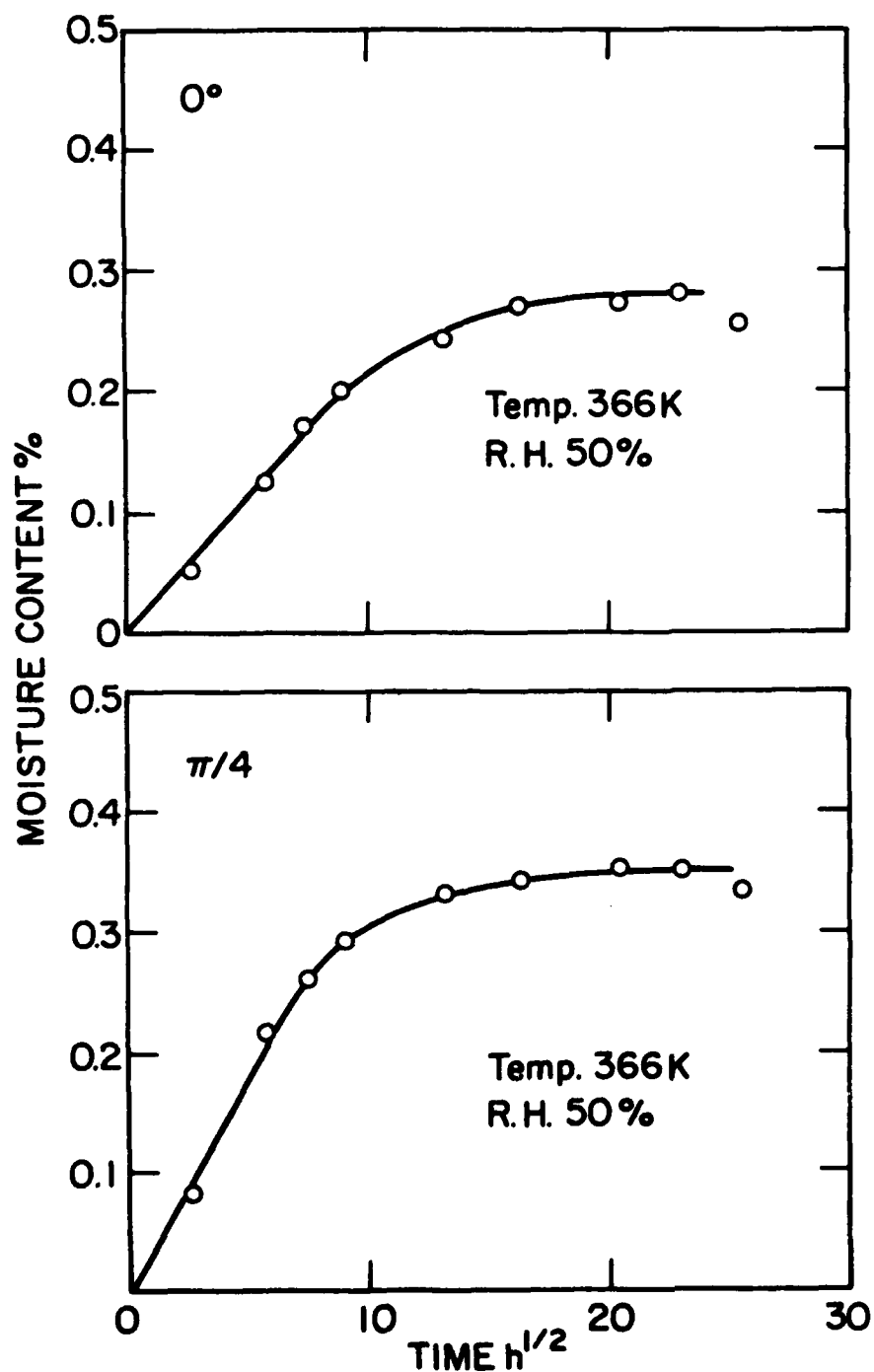
Figure B·10. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Immersed in Water at 344 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.

Figure B·11. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed
to 50% Relative Humidity at 366 K. Circle Represents
the Average of Four Data Points with the Diameter of
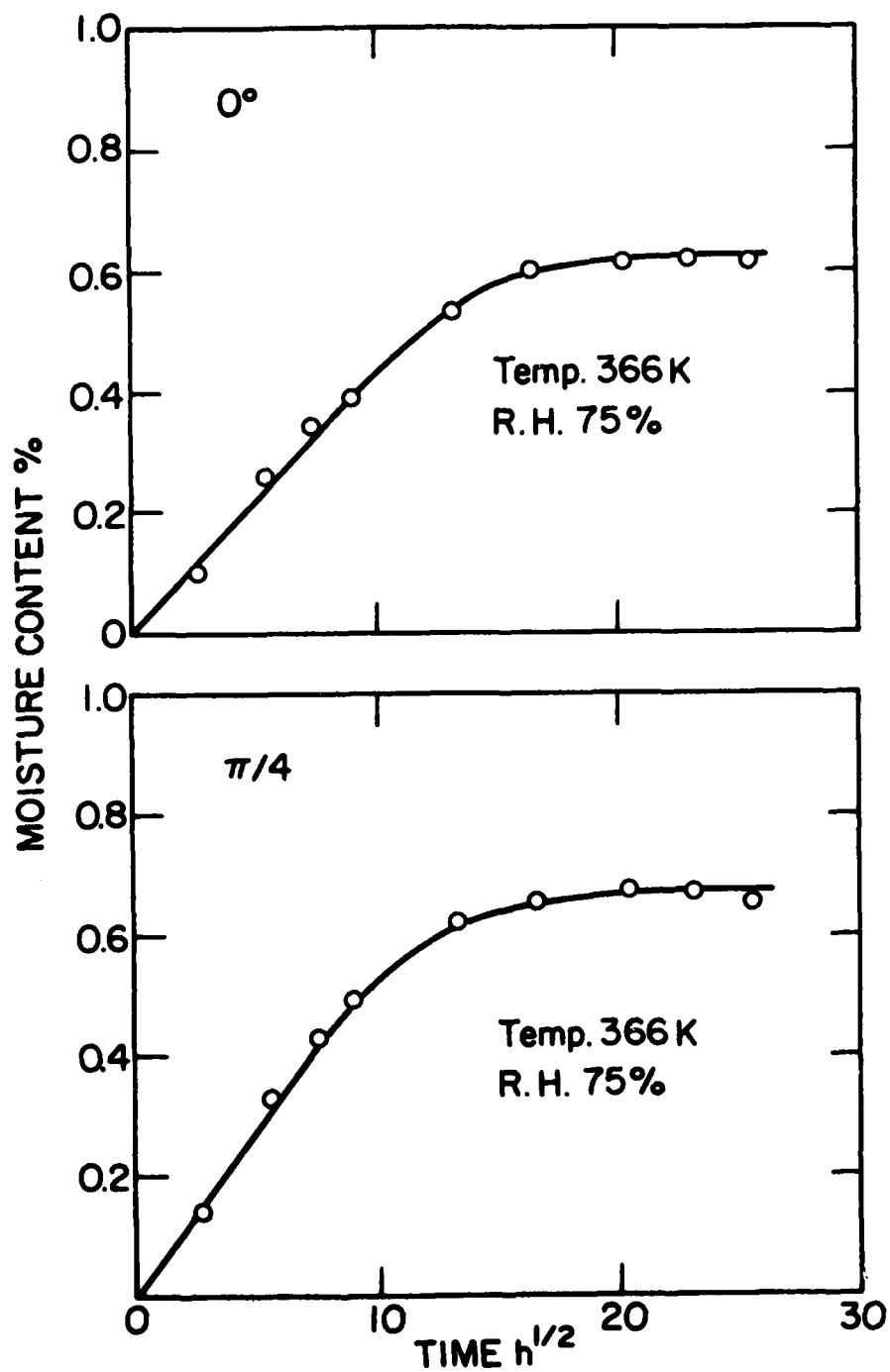Each Circle Representing the Spread in the Data. Solid
Line is Fit to Data.

Figure B·12. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed to 75% Relative Humidity at 366 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
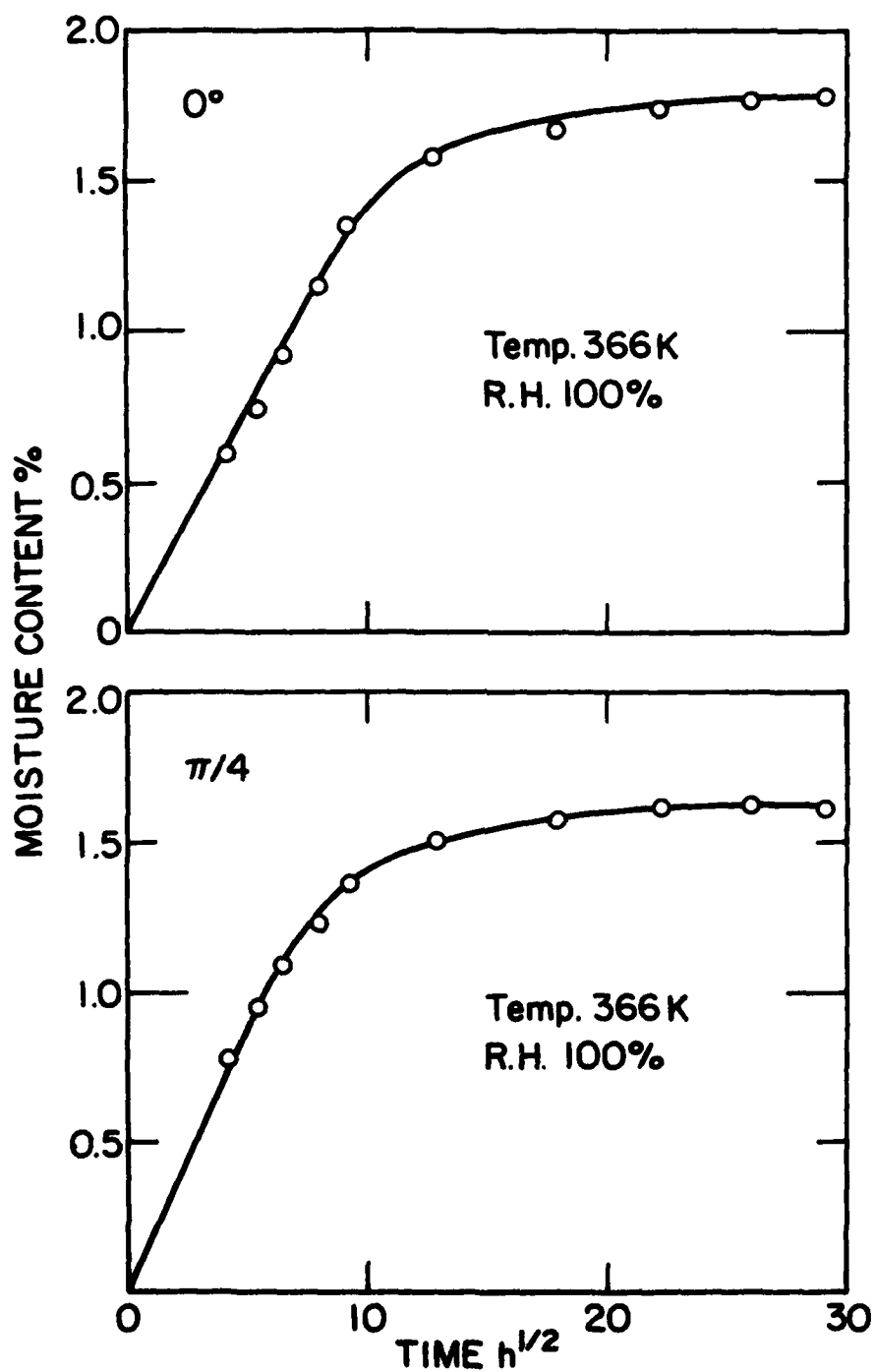
Figure B-13. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed
to 100% Relative Humidity at 366 K. Circle Represents
the Average of Four Data Points with the Diameter of
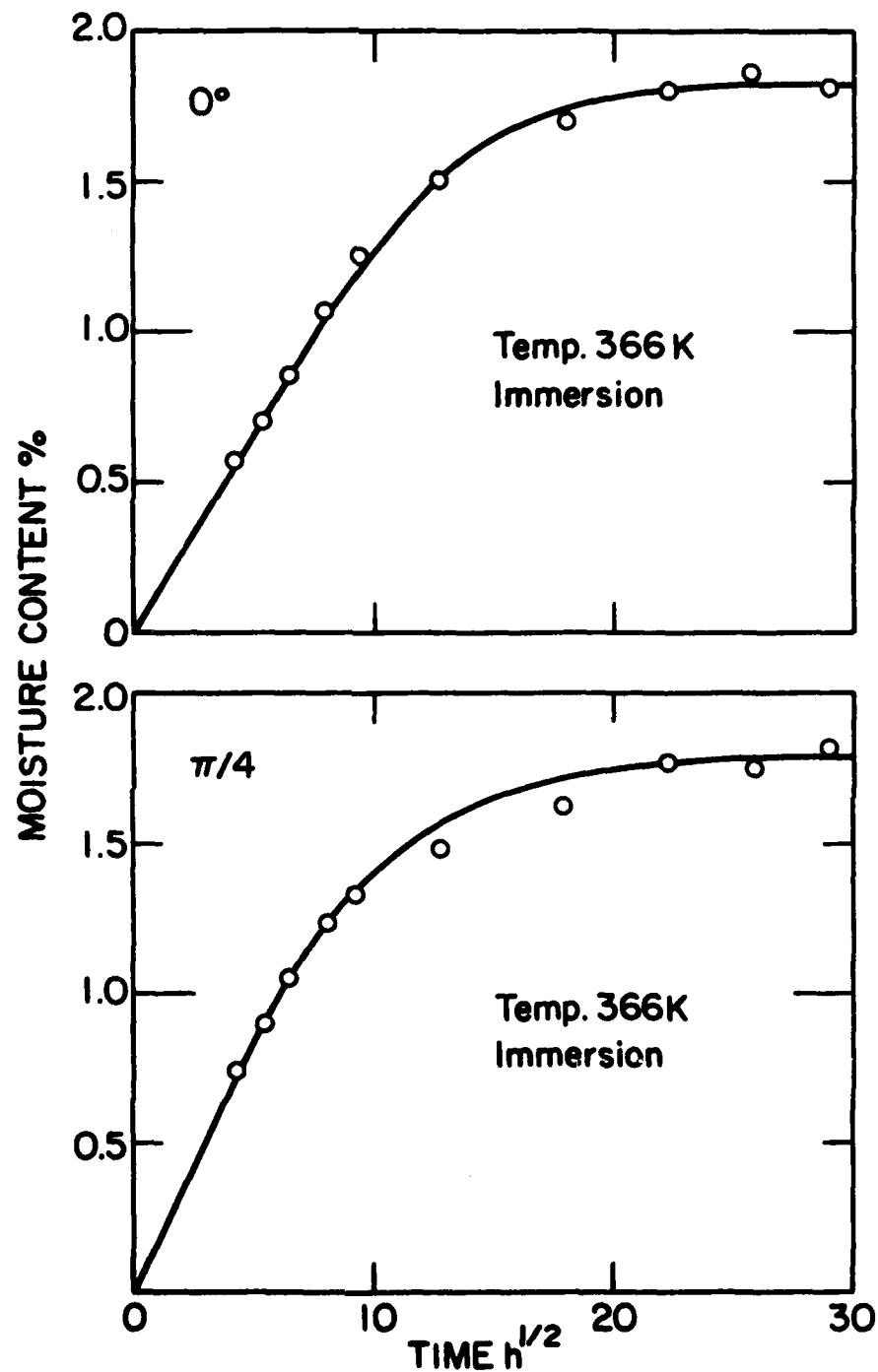Each Circle Representing the Spread in the Data. Solid
Line is Fit to Data.

Figure B·14. Moisture Content Versus Exposure Time for Unidirectional and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Immersed in Water at 366 K. Circle Represents the Average of Four Data Points with the Diameter of Each Circle Representing the Spread in the Data. Solid Line is Fit to Data.
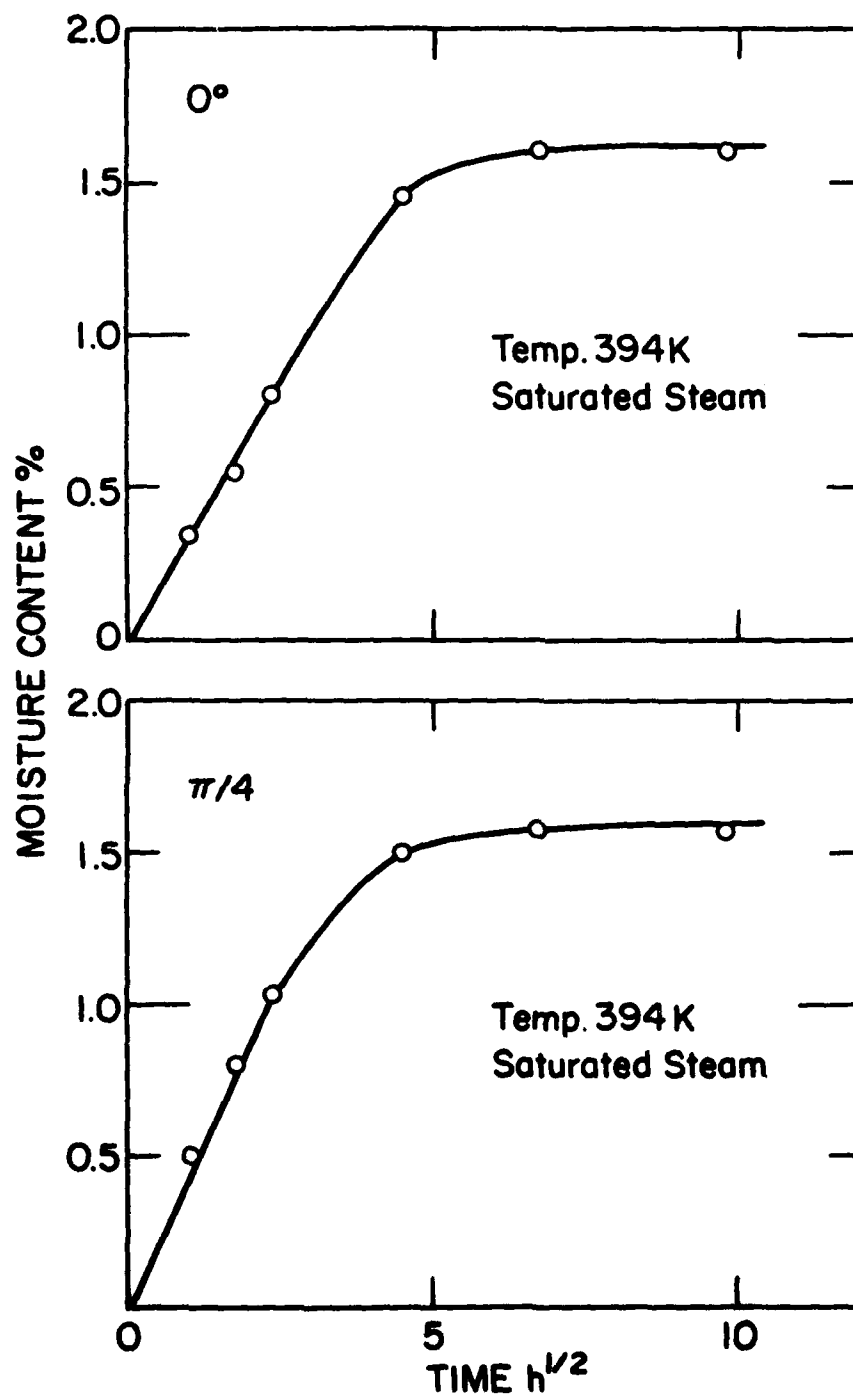
Figure B·15. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed
to Saturated Steam at 394 K. Circle Represents the
Average of Four Data Points with the Diameter of Each
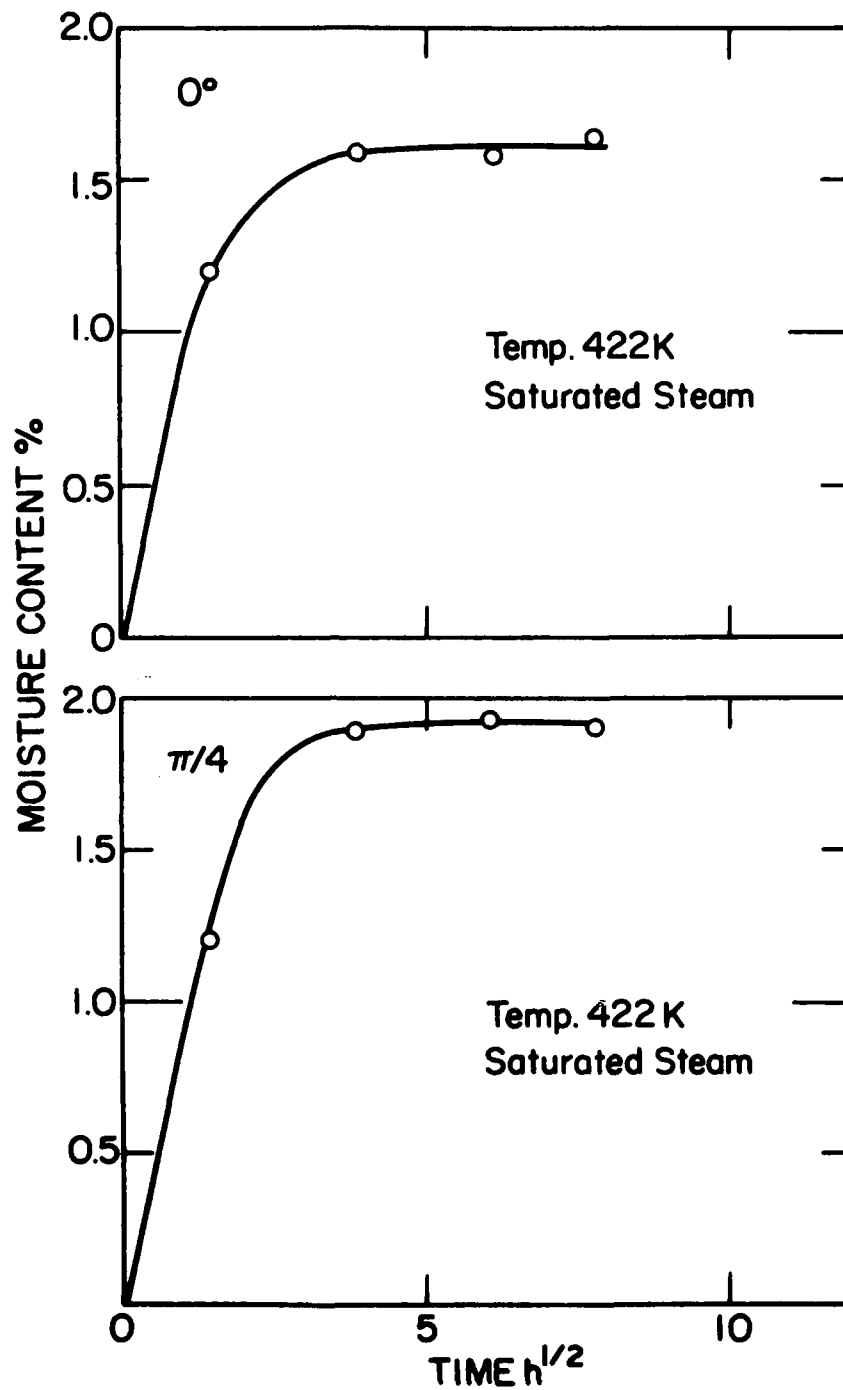Circle Representing the Spread in the Data. Solid Line
is Fit to Data.

Figure B·16.   Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Exposed
to Saturated Steam at 422 K.   Circle Represents the Average
of Four Data Points with the Diameter of Each Circle
Representing the Spread in the Data.   Solid Line is Fit
to Data.

Figure B.17. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Immersed
in Water at 422 K.   Circle Represents the Average of Four
Data Points with the Diamter of Each Circle Representing
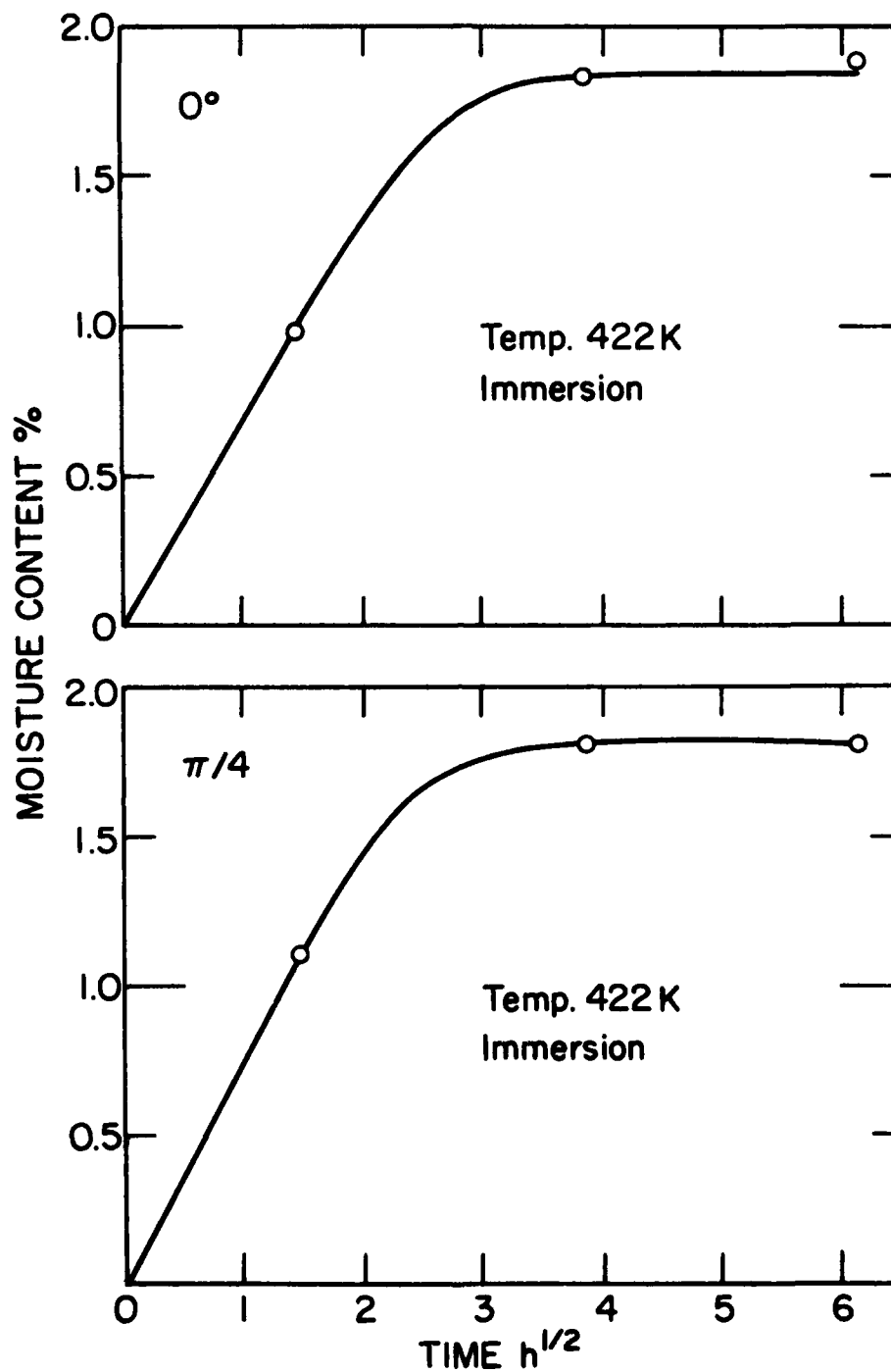the Spread in the Data.   Solid Line is Fit to Data.

Figure B·18. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Dried
at 366 K.   Circle Represents the Average of Four Data
Points with the Diameter of Each Circle Representing the
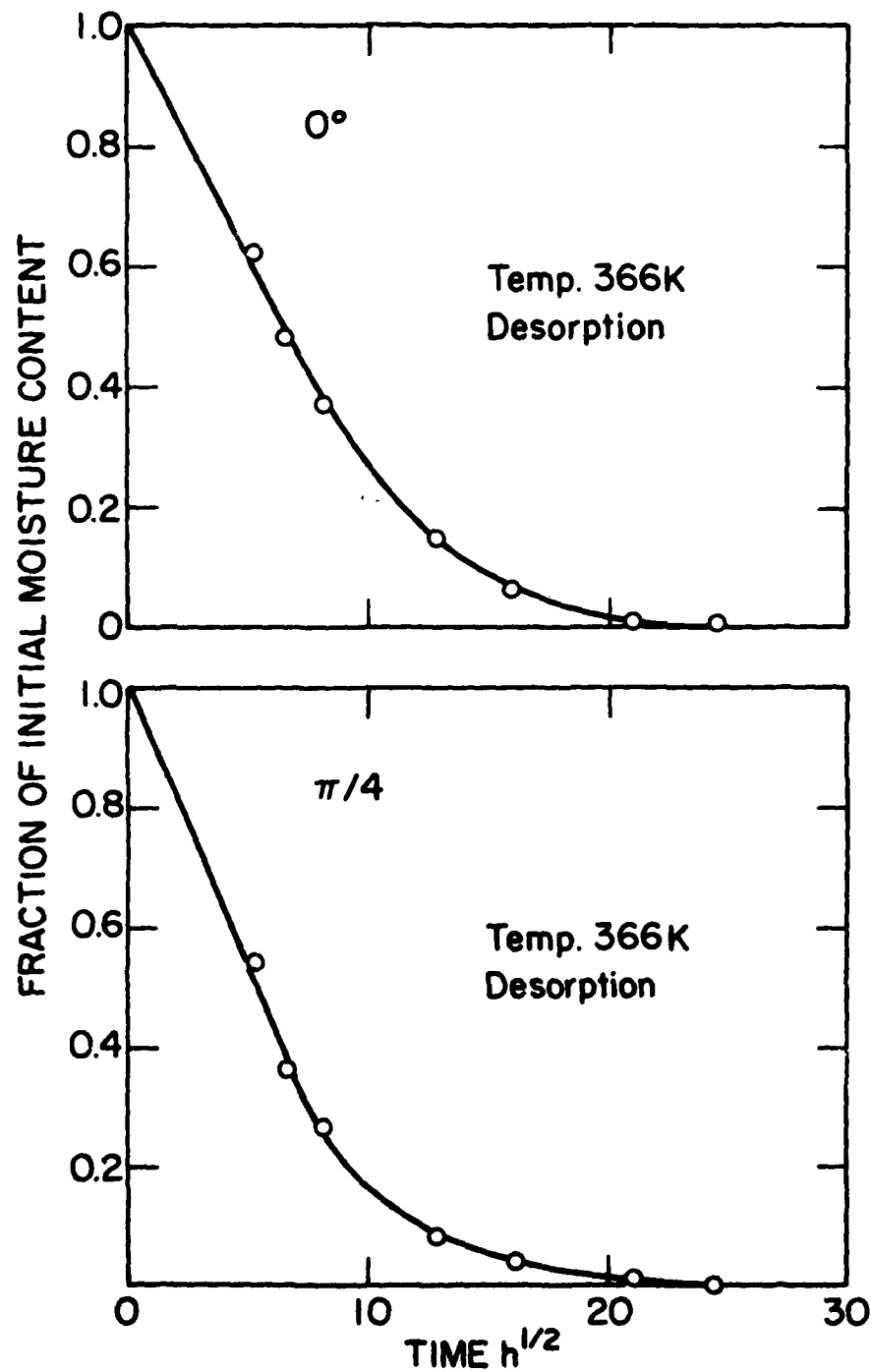Spread in the Data.   Solid Line is Fit to Data.

Figure B·19. Moisture Content Versus Exposure Time for Unidirectional
and $\pi/4$ Graphite T-300 Fiberite 1034 Composites Dried
at 422 K. Circle Represents the Average of Four Data
Points with the Diameter of Each Circle Representing the
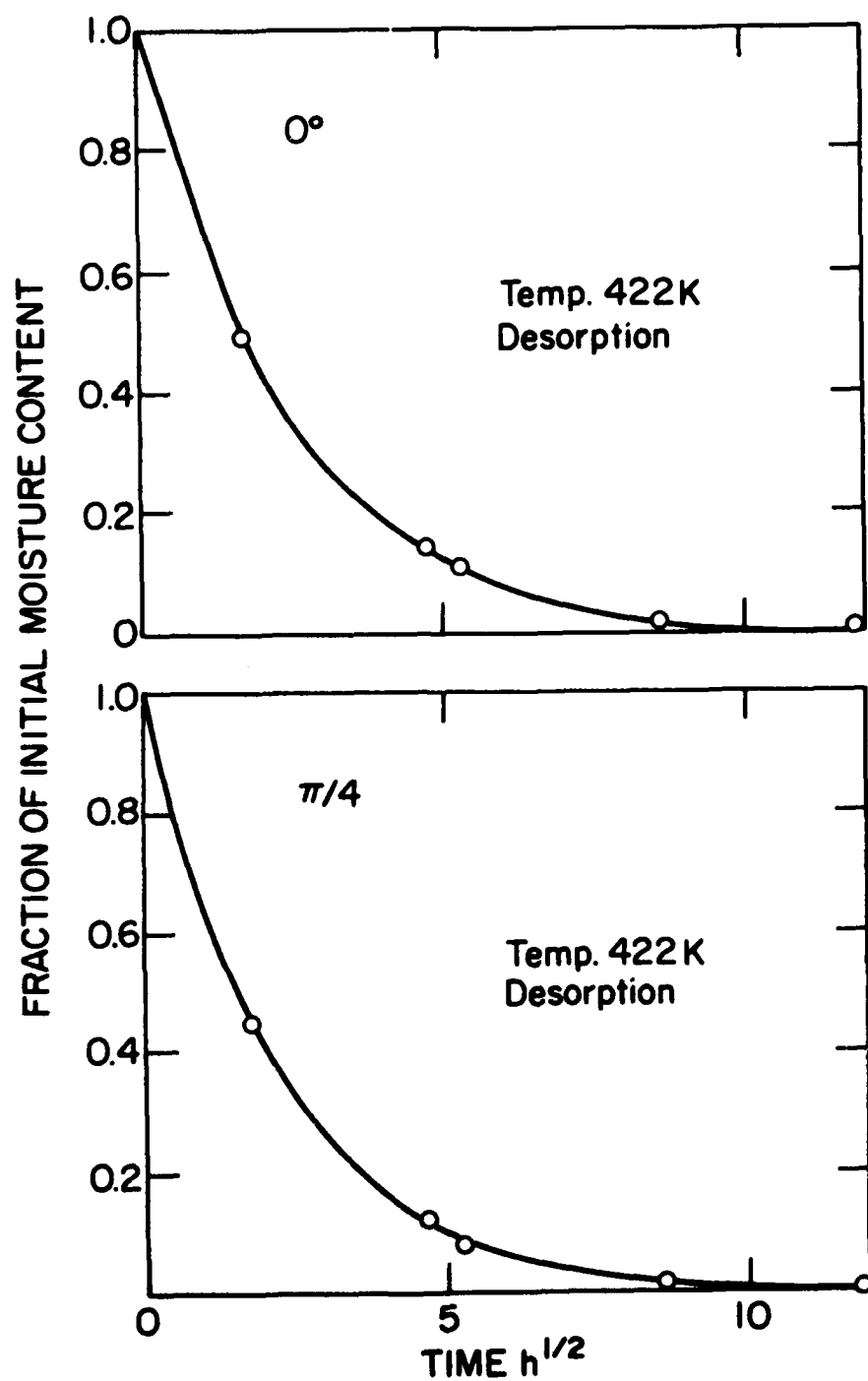Spread in the Data. Solid Line is Fit to Data.

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